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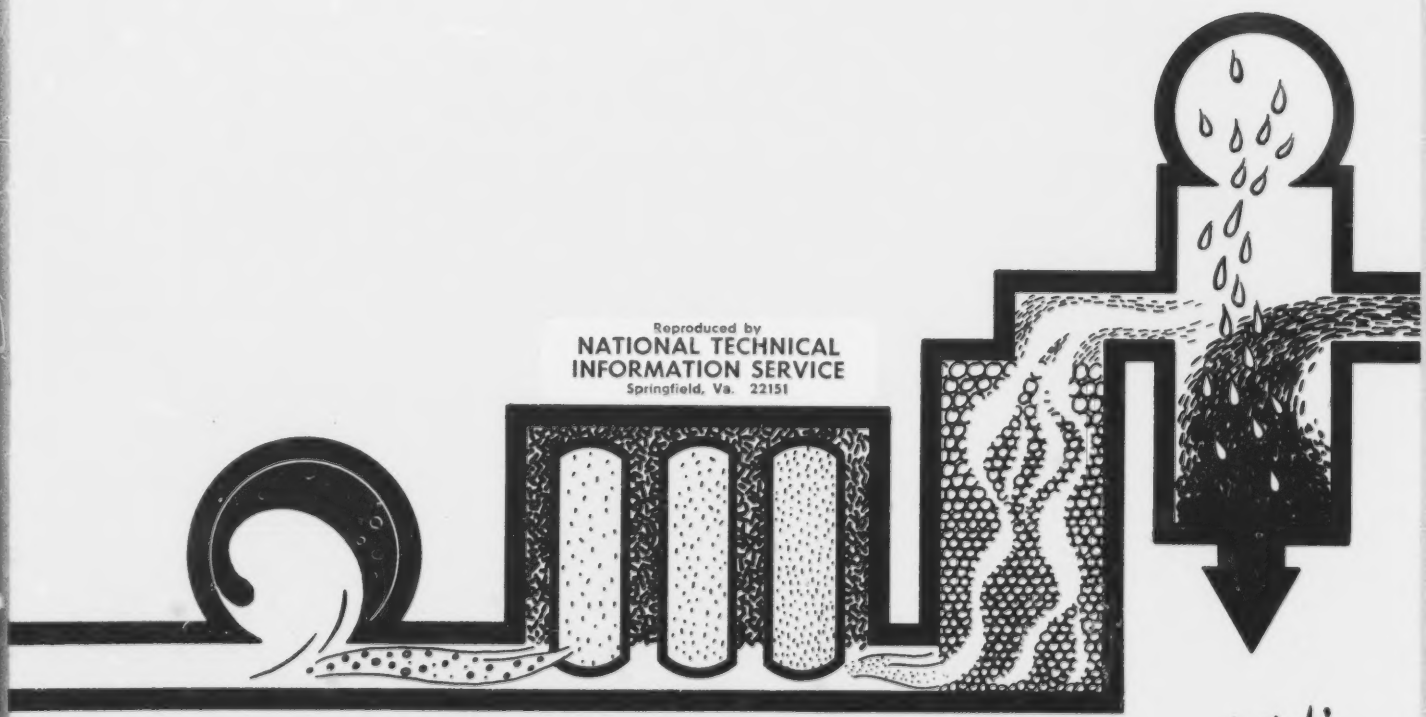

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WASTEWATER DEMINERALIZATION BY ION EXCHANGE

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for the

Office of Research and Monitoring
ENVIRONMENTAL PROTECTION AGENCY

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ABSTRACT

Pilot plant studies were conducted on sewage effluent from an activated sludge treatment plant. These studies used a pretreatment system and an ion exchange system operating in series. The pretreatment system consisted of lime clarification, dual media filtration, and granular activated carbon filtration. This system reduced the total phosphate, suspended solids (turbidity), and total organic carbon content of the wastewater as pretreatment for the ion exchange system.

The ion exchange system was the main interest of the project--reduction of the content of dissolved ionic inorganic salts in the wastewater. Conventional wastewater treatment plants do not affect the concentration of these salts. Reuse of wastewater without a reduction of these salts will cause the recycled wastewater to become unacceptably brackish.

Lime clarification will reduce phosphate concentrations; however, when ion exchange procedures are applied for wastewater demineralization, the lime clarification is unnecessary. This is because phosphate removal will occur in the ion exchange system.

Dual media filtration for removal of suspended solids followed by granular activated carbon filtration for reduction of total organic carbon is desirable.

Wastewater demineralization by ion exchange procedures can be successfully applied. Conventional ion exchange procedures are recommended. Partial demineralization can be obtained simply by applying only a weak acid cation exchange resin. This resin will reduce the total ionized solids by an amount which is equivalent to the alkalinity present in the wastewater. The reduction was approximately 50% in these studies. Demineralization of wastewater with a high concentration of nonalkaline inorganic salts requires application of two ion exchange resins: a strong acid cation exchange resin followed by weak base anion exchange resin. Operating costs can be reduced by using a third resin: a weak acid cation resin preceding (in service) the strong acid resin.

Waste regenerants from the ion exchange system must be reused, or neutralized and disposed of by locally acceptable methods. Disposal of sludges and saline liquid must be considered.

This report was submitted in fulfillment of Project 17040 EEE, contract 14-12-599, under the sponsorship of the Environmental Protection Agency.

Key words:
Wastewater demineralization, ion exchange, acid neutralization, regenerant disposal, lime clarification, activated carbon.

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SECTION 1

CONCLUSIONS

Pilot plant studies of several ion exchange systems have been conducted with a treated sewage effluent from an activated sludge plant. These studies have led to the following conclusions.

1. The phosphate content of secondary treated sewage is reduced by addition of hydrated lime (calcium hydroxide). The approximate reduction is from 18.6 mg/l (median value) to less than 2 mg/l at pH 10.0; or, to a concentration of less than 0.5 mg/l at pH 10.5.

2. The phosphate concentration can also be reduced from the 18.6 mg/l median value to less than 2 mg/l simply by ion exchange.

3. Ion exchange demineralization of this sewage which contains high alkalinity (median value of 392 mg/l as calcium carbonate) is more costly with lime clarification than it is without lime clarification. The chemical cost for the cation exchange resin partial treatment of 1000 gallons of sewage is 18¢ with lime clarification, as opposed to the lesser cost of 15.3¢/1000 gallons when lime clarification is not used.

4. Activated carbon adsorption in granular beds with a contact time of 40 minutes (empty bed basis) reduces the total organic carbon concentration of the secondary treated sewage by 40-60%, as measured by analyses for total organic carbon.

5. The ion exchange process which uses the bicarbonate form of a weak base anion exchange resin is not sufficiently established to justify pilot plant studies on sewage containing as little as 500 mg/l of dissolved ionizable solids. Operating procedures, recarbonation in particular, are indistinct and must be better established before further pilot plant work can be justified.

6. The ion exchange process using strong acid cation exchange resin and weak base anion exchange resin can be readily applied to wastewater demineralization without difficulty. Design of treatment plants for such a process applied to wastewater containing as much as 500 mg/l of ionizable solids can use established design parameters.

7. Weak acid cation exchange resin can be used very efficiently as the first resin in an ion exchange system to demineralize wastewater which contains the amount and ratio of hardness and alkalinity found at the Elgin, Illinois pilot plant.

8. Organic fouling of the ion exchange resins used did not develop.

9. Waste regenerant acid, from the cation exchange resins, was minimized by using both weak and strong acid cation exchange resins in series. The acid is easily neutralized with either limestone or hydrated lime.

10. Waste regenerant ammonium hydroxide from the anion exchange resin can be treated with hydrated lime for liberation of ammonia, which can be recovered and reused.

11. Operating costs for chemicals (regenerants and neutralizing agents) to treat the activated sludge treated sewage effluent at Elgin, Illinois range from 6.7 to 23.8 cents per 1000 gallons of product water. The low value is for partial demineralization for removal of alkaline salts by weak acid cation exchange. The high value is for complete exchange of all ionic contaminants, without regenerant recovery. A realistic cost for complete demineralization will be between these two values.

SECTION 2

RECOMMENDATIONS

1. Design of plants for wastewater demineralization by ion exchange using the demonstrated processes should be initiated. Sizes of plants should be selected by the Environmental Protection Agency. Plant design should include cost estimates for capital and operating expenses.

2. The pilot plant facilities should be maintained operating with the optimum ion exchange system for this wastewater to demonstrate the reliability of the resins' operating characteristics. Repetitive cycling through exhaustion and regeneration to demonstrate reproducibility of quantity and quality of product should be continued for an uninterrupted six months test.

3. The pilot plant facilities should be maintained in further demonstration of other ion exchange processes for wastewater demineralization. Such other processes should be established by agreement between the Environmental Protection Agency and the contractor.

SECTION 3

INTRODUCTION

Demands for increased amounts of usable water are prevalent. Satisfying these demands requires not only prudent use of water supplies, but its resue wherever practical. Wastewater has, because of its very nature, always been discarded as unusable, in spite of diminishing supplies of "fresh water". Removal of objectionable contaminants from wastewater will permit its reuse. This recycling of wastewater by its reuse will achieve two primary goals. The first of these is an increase in the available supply of water at a location of water usage. The proximity of the wastewater reclamation plant and the need for an increased water supply precludes high distribution costs. The second achievement is a reduction of pollution downstream from the reclamation plant.

Wastewater reclamation requires the removal of impurities which may be broadly classified in three categories: insoluble materials, soluble organic materials, and soluble inorganic salts. Whereas many operating plants partially remove the contaminants in the first two categories, separation of dissolved inorganic salts from wastewater has not been widely applied. Separation techniques using ion exchange demineralization are known, but their application to sewage is not generally practiced. Nor have these techniques been studied in sufficient detail in the application to wastewater to permit predictable performance.

Reduction of dissolved inorganic salts is readily obtained by the application of the ion exchange process. This process has been in wide commercial use for decades, but application to wastewater treatment has been negligible. A prime deterent has been the affect of high molecular weight organic compounds present in the wastewater. These compounds have a deleterious effect on most anion exchange minerals. Recently, new types of resins, less affected by organics, have become commercially availabe. The present investigation includes studies on the performance of these newer resins on wastewater demineralization.

Ion exchange resins must be periodically regenerated with chemical reagents. Regeneration produces a relatively small volume of a highly mineralized waste. The application of ion exchange systems for wastewater demineralization must consider reduction of these waste regenerant volumes together with their disposal. Possible reclamation and reuse of regenerants has been considered in this investigation.

The Environmental Protection Agency, Water Quality Office and Culligan International Company have concluded the agency's Contract No. 14-12-599 to study ion exchange processes applied to wastewater reclamation.

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Culligan constructed a pilot plant on the properties of the Elgin Municipal Sanitary Treatment Plant. Daily sewage flow through the municipal sewage treatment plant is approximately 6 million gallons per day during the winter months, peaking at 12 million gallons during the summer. The plant uses both trickling filter and activated sludge secondary treatment. This investigation used the effluent from the clarifier of the activated sludge effluent as the feed to the pilot plant. Most of the work for this investigation has been at the pilot plant. Detailed chemical analyses in support of the pilot plant were made at the Analytical Laboratories of Culligan International Company. Studies on possible regenerant disposal and reuse have been carried out in the Research Laboratories of the company.

SECTION 4

OBJECTIVES

This project was initiated with two main objectives which were obtained. The first was to construct a highly flexible pilot plant to study several ion exchange systems for the treatment of the effluent from secondary treated sewage.

The second objective was to observe the performance characteristics of the ion exchange systems by operating the pilot plant. Operating conditions and processes were varied in order to obtain performance data for the systems. This performance data is presented in this report for use in estimating treatment costs for wastewater demineralization by ion exchange.

Secondary objectives include an evaluation of the pretreatment processes. Also, neutralization and reuse of regenerants were studied.

SECTION 5

PILOT PLANT DESCRIPTION

The pilot plant was located on the properties of the Elgin Municipal Sanitary Treatment Plant. Activated sludge treated sewage clarified effluent was delivered to the pilot plant. The pilot plant consisted of two basic systems. The first was a pretreatment system to further treat and clarify the influent received from the activated sludge clarifier. The second was the ion exchange system to receive the effluent from the pretreatment system. Figures 1 and 2 are photographs showing the exterior of the pilot plant. The pretreatment system is housed in the frame building, while the ion exchange system is contained in the adjacent trailer.

Suitable valving and sample points, combined with indicating and recording instruments, were provided in the pilot plant to permit efficient operation and collection of adequate, accurate data.

The pilot plant included facilities, partly shown in Figure 3, for wet analyses of samples. Immediate chemical analyses and instruments' readout at the pilot plant permitted rapid decisions concerning the performance of the ion exchange and pretreatment systems. Verification of the results with greater analytical detail was obtained by subsequent analyses at the analytical laboratories of Culligan International Company.

Pretreatment equipment description. A brief description of the equipment in the pretreatment system is offered. Figure 4 shows the flow sheet, while Figure 5 shows a photographic view.

Influent to the pretreatment system was collected from the activated sludge clarifier effluent by means of a low head centrifugal pump. A flow restrictor placed in the discharge side of the pump limited the flow into the pretreatment system to 15.0 gallons per minute. This flow rate was the maximum design rate to provide 45 minutes detention in the clarifier in our pretreatment system.

A dry feeder was mounted on top of the clarifier to feed hydrated lime (calcium hydroxide) for clarification, phosphate precipitation, or other tests as dictated by the test program. The feed rate was controlled by a pH indicator - controller which analyzed a small sample stream collected from the bottom of the rapid mixed chamber of the clarifier. The pH indicator - controller had adjustable set points to permit fine control of the pH.

The clarifier tank was cylindrical, 6 feet in diameter, 7 feet tall. Flow was bi-directional: downwardly in the center of the tank, through

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Figure 1 - Photograph of Pilot Plant front view, showing ion exchange systems trailer against pre-treatment systems shelter.

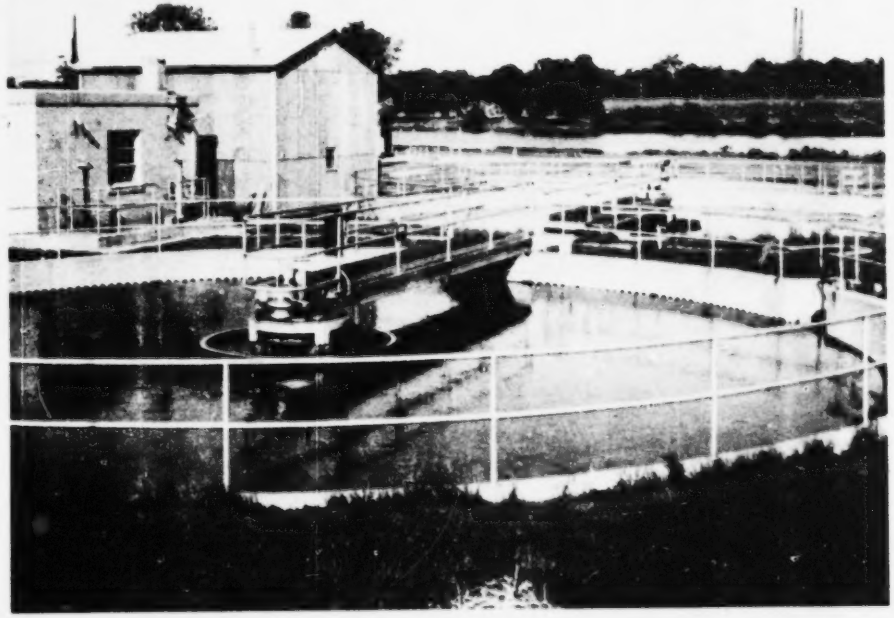

Figure 2 - Photograph of Pilot Plant rear view, showing activated sludge clarifiers which supply influent for tests.

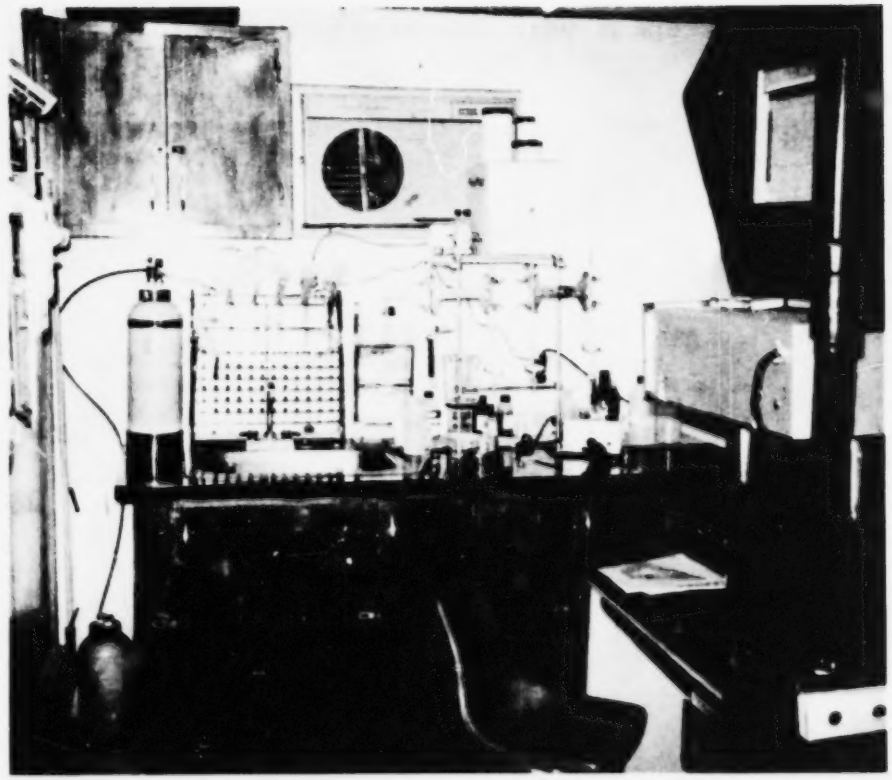

Figure 3 - Photograph of Pilot Plant wet analysis laboratory.

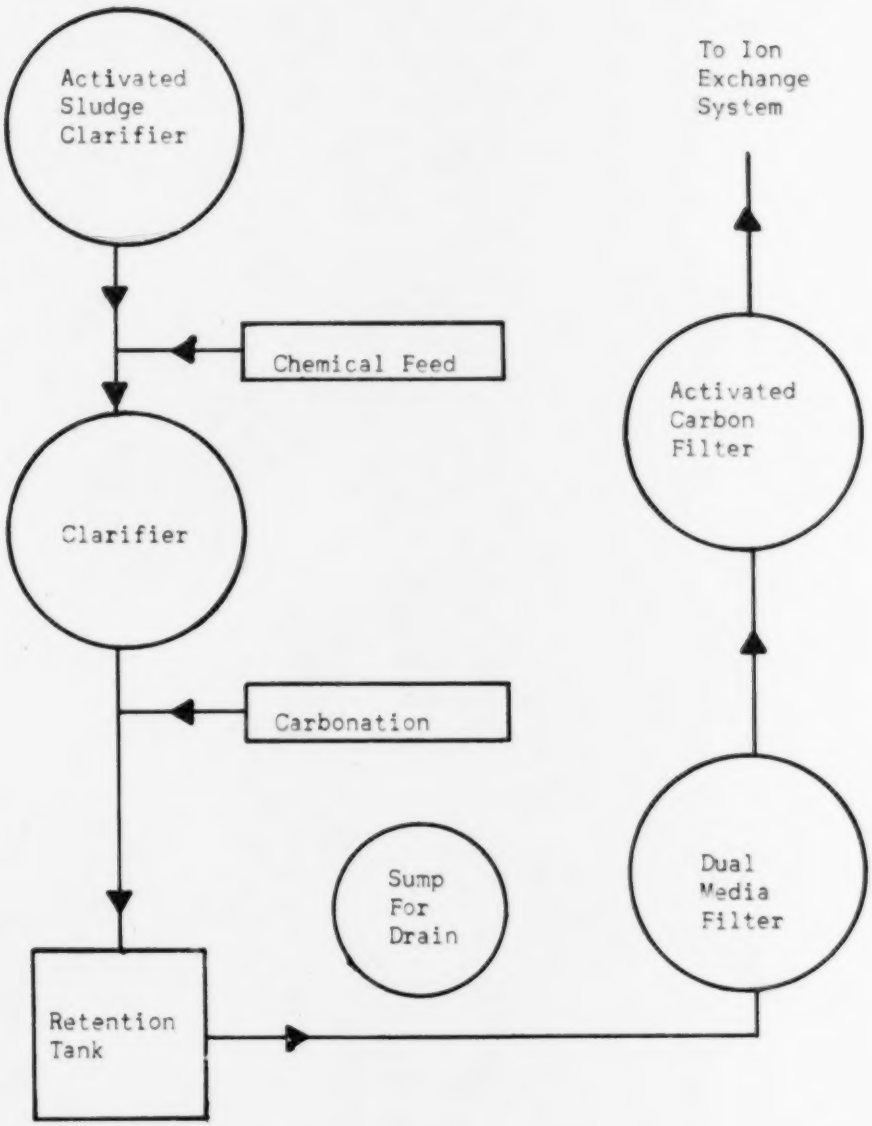

Figure 4. Pilot Plant Pretreatment Flow Sheet

Figure 5 - Photograph of Pilot Plant pretreatment systems.

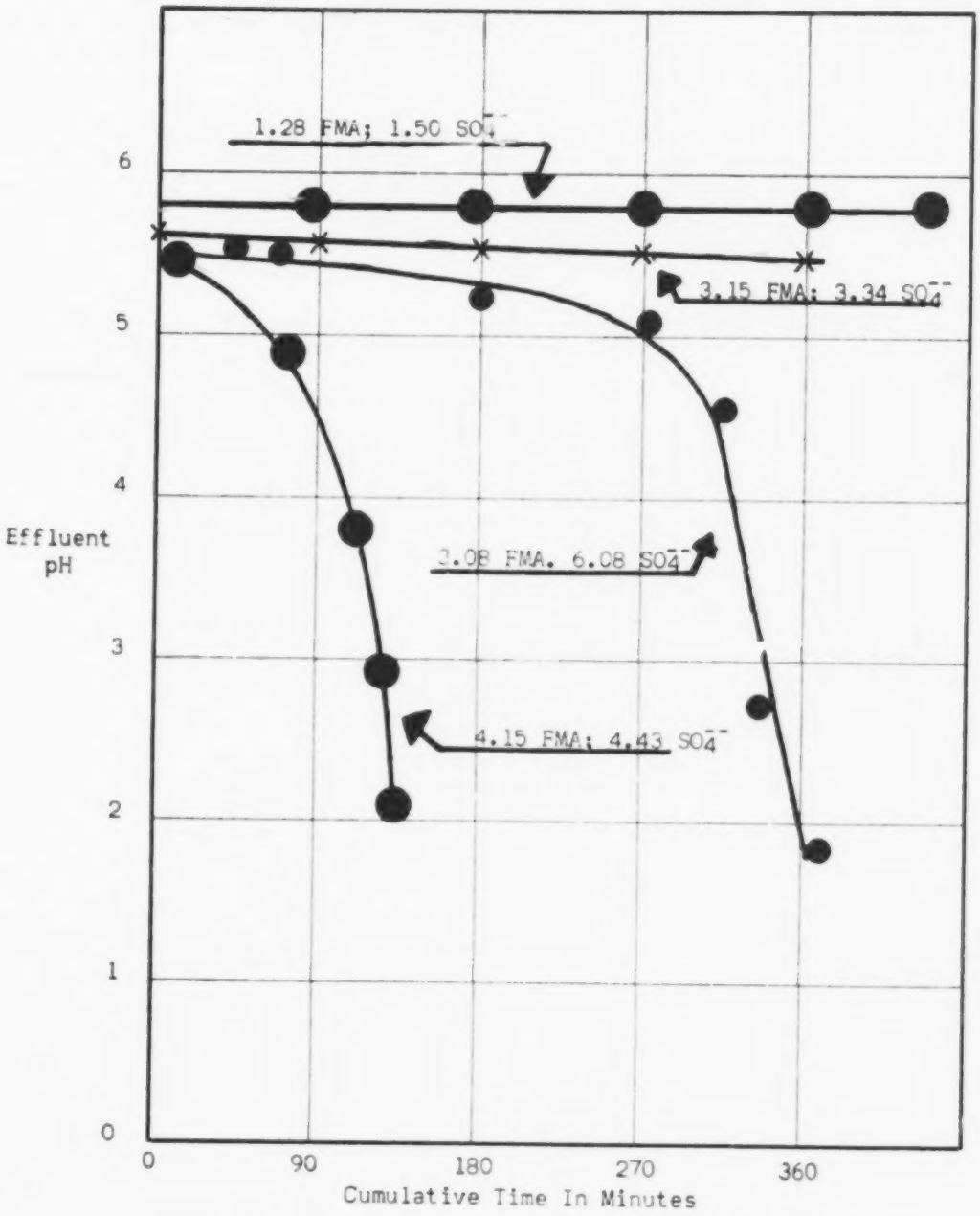

Figure 19. Dynamic neutralization of waste acid. Upflow through 36 inches of limestone with 45% bed expansion at 70° F. Influent FMA and sulfate, in grams per liter as $CaCO_3$, shown on curves; for complete analyses see Table 44.

Ion exchange equipment description. The equipment was housed in the mobile truck trailer shown in Figures 1 and 2. Figure 6 shows a flow diagram of this equipment. The versatility of the ion exchange system is illustrated in Figure 7, showing the piping and valve assemblies located in front of the ion exchange vessels.

Easy manipulation of the valves in the ion exchange system to accomplish the desired flow path was readily obtained by the pilot valves on the control panel illustrated in Figure 8. Operation of a pilot valve illuminated an indicator light on the graphic panel mounted above the control valve assembly. Therefore, the status of each valve was readily known.

The ion exchange system consisted of five pressure vessels, containing ion exchange resin. These vessels ranged from 10 to 14 inches in diameter, 48 to 72 inches in height. The smallest vessel accomodated $1\frac{1}{2}$ cubic feet of resin with a 33 inch bed height, while still allowing an approximate 30% freeboard. The pressure vessels were operated individually, or in various combinations. Flow direction during service or regeneration, through the vessels was upflow or downflow as desired.

Regeneration was accomplished by preparing a solution of the desired regenerant in a separate open vessel. A pump transferred this solution through the proper valves operated at the valve control panel to the ion exchange vessels. Rinse-out of the regenerant was accomplished by pumping rinse water from the regenerant vessel, or by use of selected water streams from the plumbing network of the ion exchange system. The usual procedure was to use both sources of rinse water - a small quantity of softened potable water following the regenerant injection so that interconnecting plumbing was flushed; final regenerant rinse-out was accomplished with "system water".

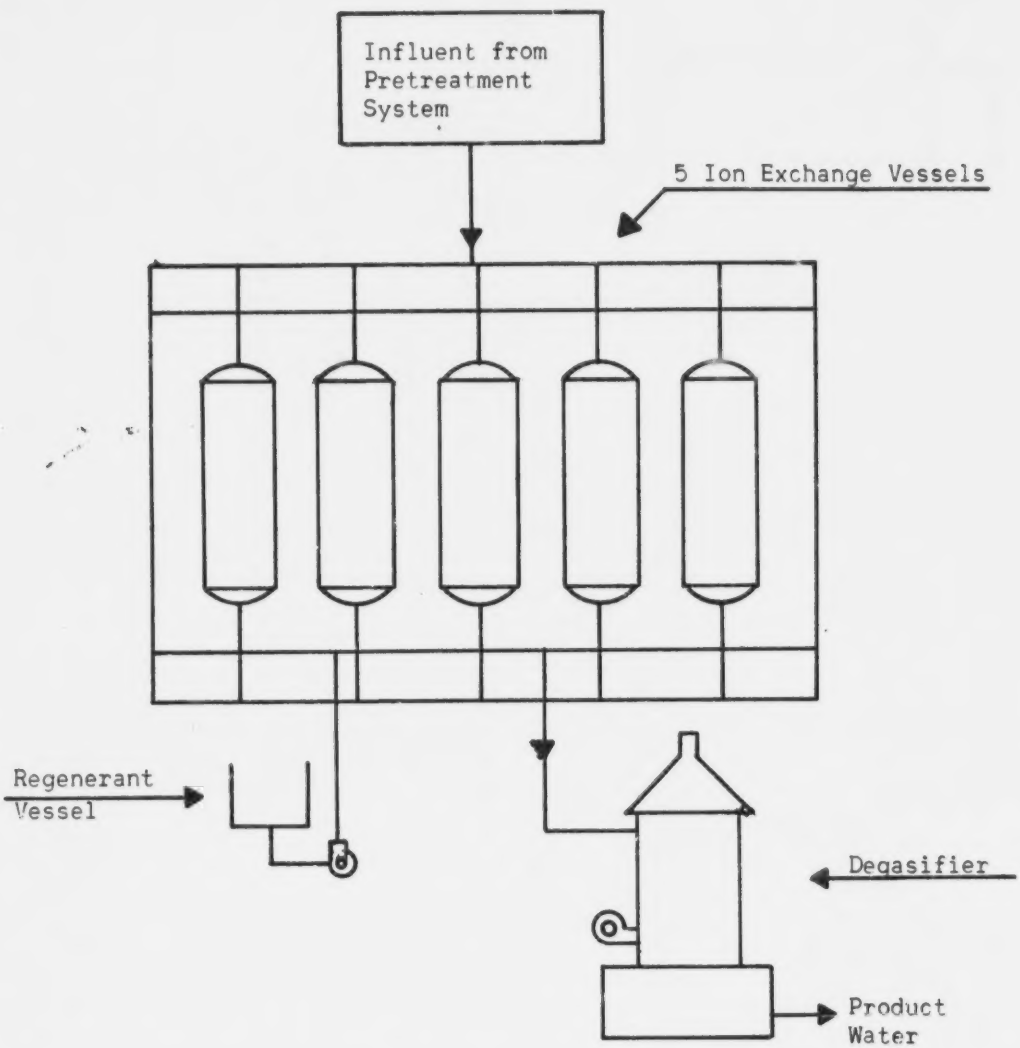

Figure 6. Pilot Plant Ion Exchange Flow Sheet

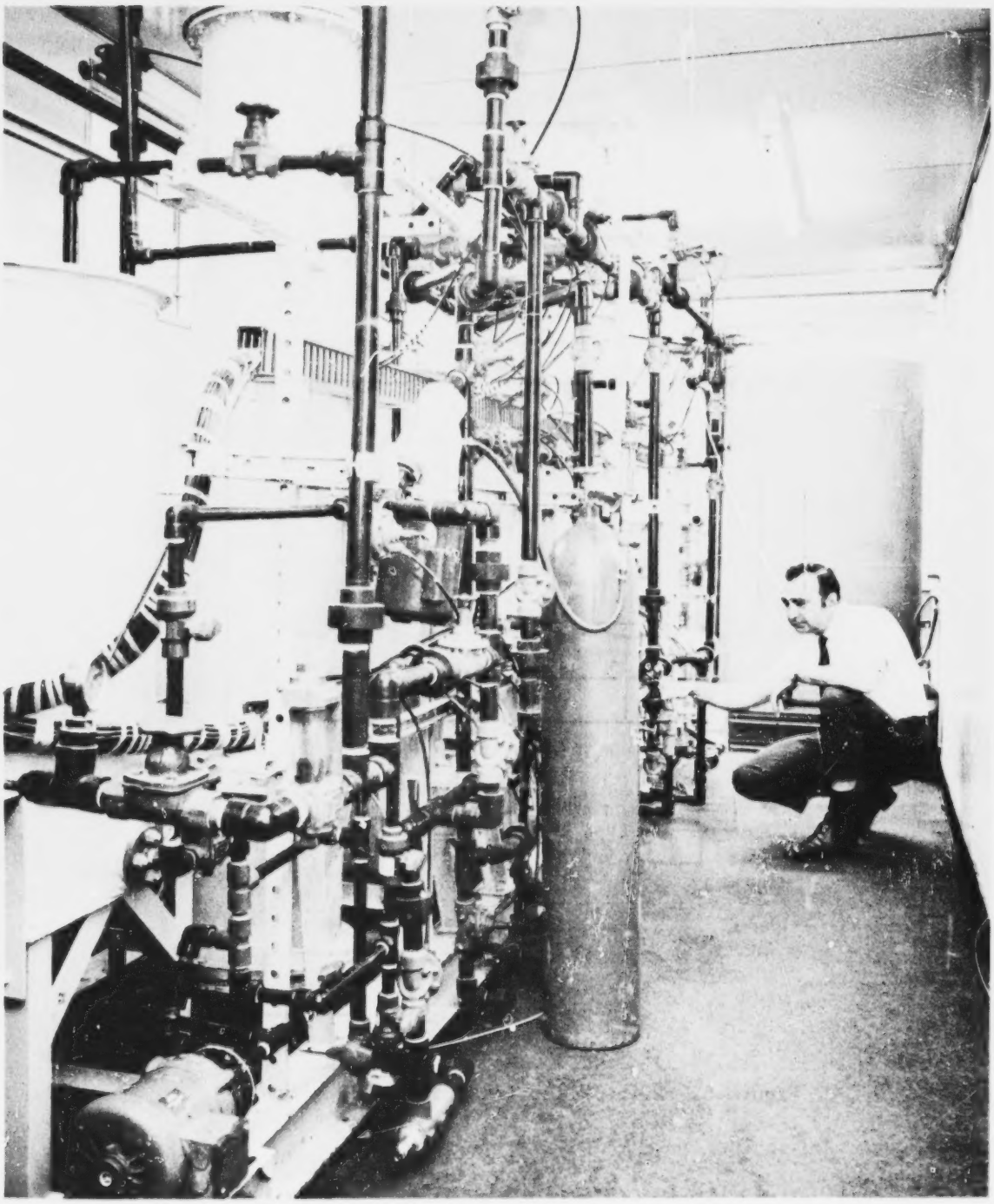

Figure 7 - Photograph of Pilot Plant ion exchange systems, showing piping versatility - vessels located behind piping.

Figure 8 - Photograph of Pilot Plant ion exchange valve control panel.

SECTION 6

PRETREATMENT OPERATION

During the first four months of this project, the attention given to the performance of the pretreatment system (treatment of the influent treated sewage preparatory for ion exchange treatment) was more detailed than during the remainder of the project. Later studies did not use the clarifier and recarbonation processes.

Throughout the project, wide variations were encountered in the raw water chemical composition. The natural variations of the potable water supply to the surrounding communities were further varied by the diverse use of this water before discharge to the sewer. Surface run-off with its natural contaminants plus street deicing salt during the winter months were further causes of variation.

Frequent detailed chemical analyses were made of our influent from the sewage plant during the first four months. Later studies were to a lesser detail. Table 1 shows the variations which we encountered in the influent water. The table shows the low, high, and median values of our analyses for the constituents of our influent received from the activated sludge clarifier. Table 1 also shows the percent maximum deviation from the median valve. Largest deviations are to be found in the nitrogen constituents (ammonia, nitrate, and nitrite) and in the total organic carbon. These large variations indicate variable operating efficiency of the activated sludge process at this location. Repeated variation in our influent water continued throughout the project.

This influent was pumped to our clarifier as discussed in Section 5. The clarifier was designed to treat 15 gallons per minute, which provided 45 minutes retention with a rise rate of 0.57 gallons per square foot per minute. Operational difficulties were encountered with the clarifier due to non-continuous operation. The clarifier was shut down each night. Therefore, on the following morning, some time was required to stabilize the clarifier operation. During this stabilization time, the clarifier effluent was diverted to drain; while stored, recarbonated, water in the retention tank was used for ion exchange system tests.

Our clarifier operated with lime (calcium hydroxide) as a precipitant. A volumetric dry feeder was mounted on top of the clarifier for controlled addition of the lime. Jar tests were first used to establish the desired dosage rate. Figures 9, 10, 11 show the effects produced in these jar tests by varied doses of lime. A lime dose of 500-600 mg/l produced a pH of 10-11, and turbidity of 4-5 JTU, while reducing the total phosphate (as PO_4^{---}) from 10-12 to less than 1 mg/l.

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Table 1. Variation of Influent Wastewater, Which Was Received From Activated Sludge Clarifier.

Constituent*	Low Value	High Value	Median Value	Deviation %**
pH	7.3	8.5	7.8	--
Turbidity, JTU	8	37	22	+68
Total Alkalinity $CaCO_3$	180	452	392	-49
Chlorides, $CaCO_3$	128	226	163	+39
Sulfates $CaCO_3$	91	144	105	+37
Phosphates, Total PO_4^{---}	6.2	25.8	18.6	-67
Magnesium, $CaCO_3$	99	222	196	-50
Calcium, $CaCO_3$	76	229	120	+91
Ammonia, N	1.0	24.0	17.5	-94
Nitrate, N	0.1	3.7	1.5	+147
Nitrite, N	0.01	1.6	0.04	+Vast
Sodium, $CaCO_3$	198	430	273	+58
Potassium, $CaCO_3$	19	25	20	+25
Total Organic Carbon, C	4	100	39	+157

*Constituents reported in mg/l, except for pH and turbidity which are expressed in units.

**Deviation shown in percent maximum from the median value.

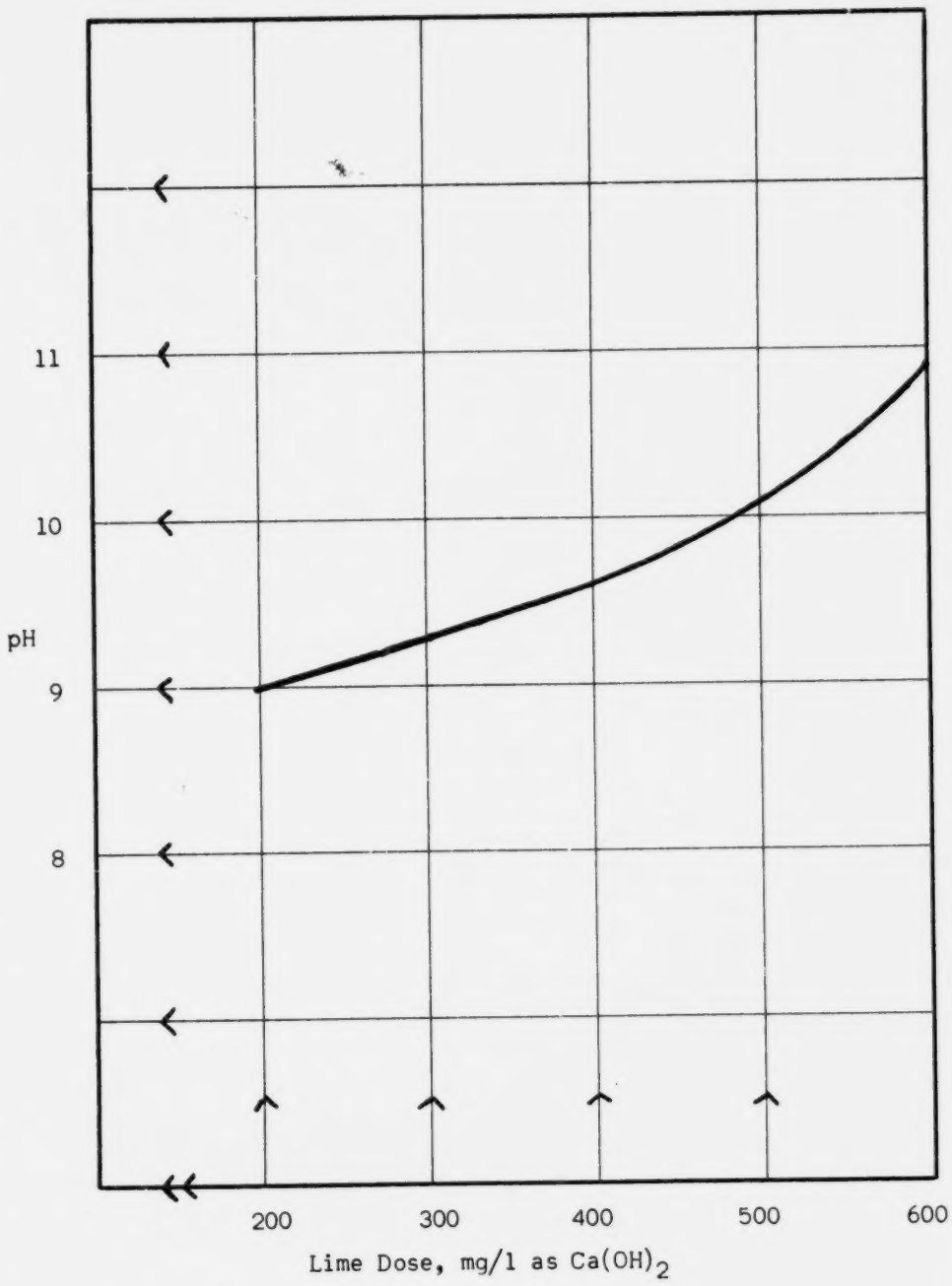

Figure 9. pH of secondary treated sewage as affected by lime. Initial pH 8.0.

Figure 10. Turbidity of secondary treated sewage as affected by lime. Settled 80 minutes.

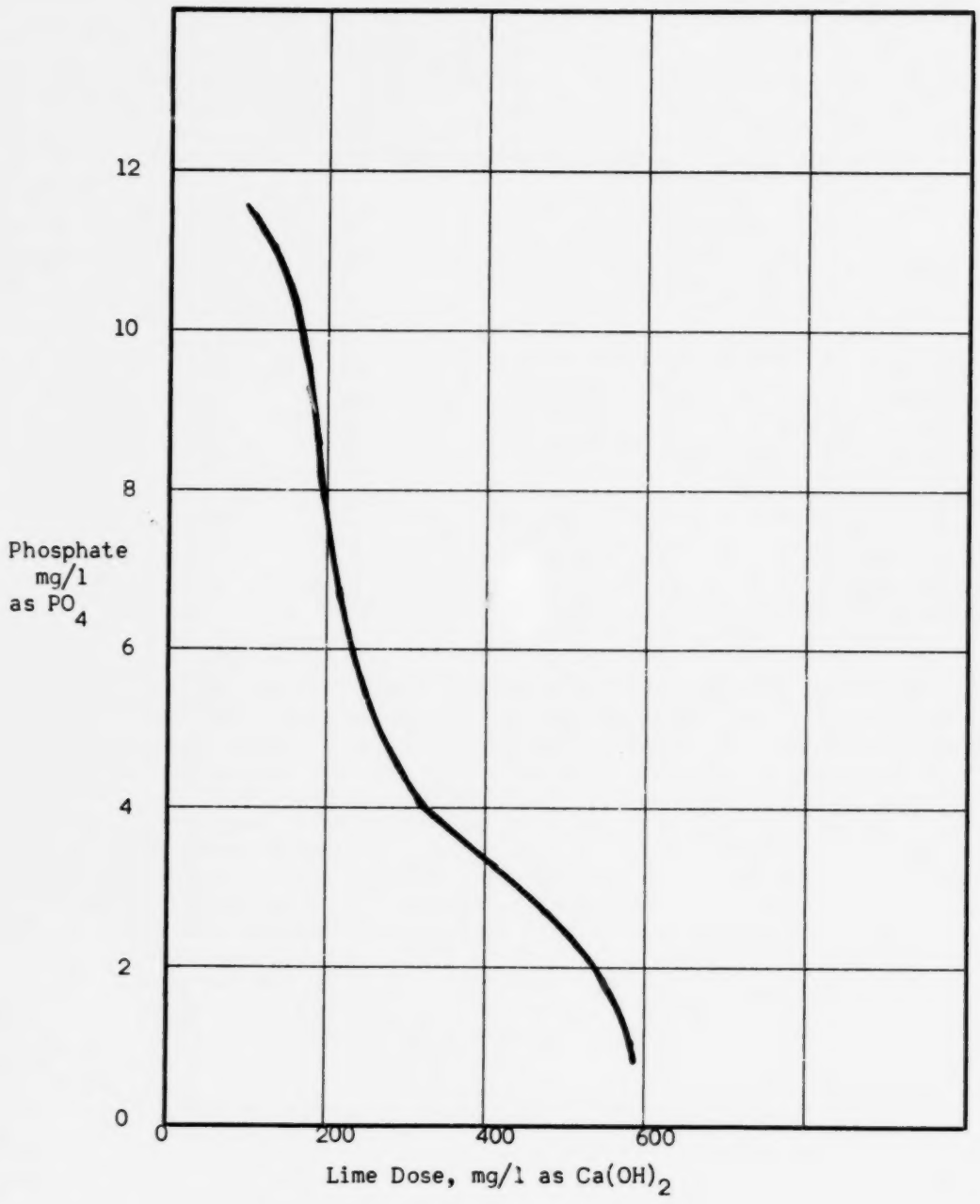

Figure 11. Phosphate concentration in secondary treated sewage as affected by lime.

The clarifier was operated in this dosage range (500 - 600 mg/l), controlled by a pH controller recorder. The intermittent operation of the clarifier resulted in turbidities greater than expected: typically, 6 JTU turbidity, with normal maximum of about 15 JTU. Similarly, reduction of phosphate was also less than expected: typically, 1 - 2 mg/l of phosphate in the effluent, with normal maximum of about 3 mg/l. Table 2 summarizes the clarifier operation. Shown are typical results at three pH levels. It must be remembered that variable raw water composition makes comparison of these results difficult.

Table 2. Clarifier operation - typical effluent analyses.

pH, units	10.1	10.3	10.5
Alkalinity, mg/l $CaCO_3$:			
Hydrate	0	26	3.0
Carbonate	178	125	64
Turbidity, JTU units	8	6	4
Phosphate, total mg/l PO_4	1.9	1.8	0.3
Magnesium, mg/l $CaCO_3$	89	35	20
Calcium, mg/l $CaCO_3$	69	100	84
Ammonia, mg/l N	13.1	11.2	16.5
Nitrate, mg/l N	2.8	2.1	1.4
Nitrite, mg/l N	0.13	0.04	0.08
Total organic carbon, mg/l C	35	21	16

After clarification, recarbonation with gaseous carbon dioxide was accomplished in the first compartment of the retention tank. Carbon dioxide was introduced into the downflow stream of water, while effluent pH was monitored with a recording pH meter. The pH after recarbonation was easily maintained at the desired 1 pH unit depression. Some precipitates settled in the retention tank, but these were inconsequential. The water was then filtered through a dual media filter at a design flow of 3.1 gpm/ sq ft; thence thru a granular activated carbon filter, which provided a 40 minute contact time (empty bed basis). The activated carbon filter reduced the influent total organic carbon approximately 50%. The removal of total organic carbon during the first 227 bed volumes through the filter is summarized in Table 3. When the filter was removed from service after treating 400 approximate bed volumes, reduction of total organic carbon was still effective - the remaining fraction (C/C_o) ranged 0.50 - 0.60.

Table 3. Performance of activated carbon for removal of total organic carbon, mg/l C.

Bed Volumes Treated	Total organic carbon mean mg/l C		Remaining Fraction
	Influent (C_o)	Effluent (C)	(C/C_o)
0-49	73.0	21.0	0.34
50-99	11.8	5.1	0.43
100-149	20.0	6.6	0.48
150-199	11.4	6.1	0.54
200-227	14.4	5.8	0.41

The effluent of the activated carbon filter was the influent to the ion exchange treatment system.

SECTION 7

WEAK BASE ION EXCHANGE RESIN PERFORMANCE - BICARBONATE FORM

This process employs the unusual charcteristics of a unique weak base anion exchange resin, Amberlite IRA-68. In the free base form, this resin (represented as R-N in the first equation below) is capable of adsorbing carbonic acid to form the bicarbonate salt by exposure to a solution of carbon dioxide. This bicarbonate form has a bicarbonate - mineral acidity selectivity such that neutral salts in water can be converted to alkaline salts. For example, the chlorides and sulfates of sodium, calcium, and magnesium are converted to the corresponding bicarbonate salts, as illustrated by the equation employing sodium chloride:

$$(R\text{-}NH)HCO_3 + NaCl = (R\text{-}NH)Cl + NaHCO_3$$

The effluent water, which has been treated by the bicarbonate form resin, can then be treated with a bed of weak acid cation exchange resin. This second bed of resin will convert the alkaline salts to water and carbon dioxide, as illustrated by the equation employing sodium bicarbonate:

$$RCOOH + NaHCO_3 = RCOONa + H_2CO_3$$

$$H_2CO_3 = H_2O + CO_2$$

A combination of the two ion exchange beds will then remove the ionic salts from the water, leaving carbon dioxide in solution. The latter is a gas which is readily eliminated through established technology with aeration or degasification.

Incorporation of such an ion exchange system after an efficient process for removal of organic contaminants will result in a reclaimed water without pollution due to inorganic and organic materials.

Attempts to apply the bicarbonate form of the weak base anion exchange resin have been only partially successful. The technology for the application of this process is insufficiently developed to support pilot plant studies on wastewater with low salinity. Acceptable techniques for converting the resin to the bicarbonate form were not established. Several consultations with the manufacturer of the resin were unrewarding. However, it was mutually agreed that our pilot plant techniques were proper. It is apparent, therefore, that studies of a more fundamental nature must be made on the process if it is to be applied to low salinity wastewaters.

As specified in the research contract, a bed of the weak base anion exchange resin was employed to treat the effluent from the complete pretreatment system (lime clarification, recarbonation and settling,

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filtration, activated carbon adsorption). Two cubic feet of anion exchange resin (IRA-68) were contained in a 12-inch diameter tank: the height of mineral was about 31 inches in place. Typically the resin was regenerated with a 4% solution of ammonium hydroxide at dosages which exceeded those recommended by the resin manufacturer.

Softened potable water was used for preparation of regenerant solutions and for regenerant rinse. Soft water is necessary to prevent the alkaline regenerant from precipitating the hardness cations of the raw water.

Conversion of the free base resin to the bicarbonate form was accomplished by recirculating softened water saturated with dissolved carbon dioxide, maintained at a pressure of 75 psi in accordance with the resin manufacturer's recommendations. Carbon dioxide was supplied from a tank of the compressed gas, fitted with standard regulating devices, pressure gauges, and a flow indicator. The gas was introduced into the system through an in-line carbon dioxide saturator in the pressurized recirculated water line flowing through the resin. The conditions encountered during carbonation were inexplicable. The rate and duration of carbon dioxide addition were inconsistent. No well defined means were available to determine when sufficient carbon dioxide had been used. Carbonation times varied from 1/2 to two hours to apply the calculated amount of carbon dioxide required.

Exhaustion of the bicarbonate form resin was with the sewage effluent from the pretreatment system. The exhaustion flow rate was 2 gpm/cu ft, in accordance with the resin manufacturer's recommendation and the research contract.

Termination of the resin's exhaustion cycle was determined in the field by analyses of the alkalinity and chloride in the effluent water. During service, all non-alkaline anions, such as chloride and sulfate, were exchanged for alkalinity. As exhaustion of the resin approached, complete removal of non-alkaline anions no longer occurred; as a result, the alkalinity in the effluent correspondingly decreased. Service exhaustion was terminated when the effluent alkalinity decreased about 10% from the typical value obtained on similar samples analyzed through the service cycle. Confirming chemical analyses were made on selected samples submitted to the Analytical Laboratory of the parent company. Similar field and laboratory analyses were made for chloride in the effluent. During most of the service cycle, the effluent chloride concentration was low. As the resin approached exhaustion, the effluent chloride concentration increased while the effluent alkalinity correspondingly decreased.

To determine the capacity of the resin, the influent loading factor must be known. Determination of the loading factor was made by using a small ion exchange column filled with hydrogen form strong acid

cation exchange resin. Passage of water through this column removes all cations by hydrogen exchange: acids result in the effluent. These acids are of two types: strong acids produced essentially from chlorides and sulfates; and weak acids produced essentially from the alkaline salt. The strongly acidic anions are those which must be removed in the pilot plant by the bicarbonate form anion exchange resin: the weakly acidic anions are unaffected by the bicarbonate form resin. The concentration of strong acids produced by the column was easily measured by titration with a standard solution of sodium hydroxide.

Proper sample preparation through the cation resin column requires pre-rinsing the column with demineralized water, passage of a measured sample through the column, and its rinse-out with demineralized water. The rinse water must be combined with the effluent collected from the sample passage to insure total recovery of all strong acids produced in the test column. The titration is reported in terms of calcium carbonate equivalents. This is the loading factor for calculating the anion exchange resin's operating capacity. The total operating capacity of the anion resin was then determined from the loading factor and the volume of water treated during service. For example, Table 4 attached, shows (on a per cubic foot basis) that run number one treated 86 bed volumes of water with a loading factor of 340 mg/l. The 86 bed volumes is 645 gallons (each cubic foot has a bed volume of 7.5 gallons) per cubic foot of ion exchange resin. The loading factor of 340 mg/l is 19.9 grains (340 mg/l divided by 17.1) of loading per gallon of water treated. Multiplying these two products results in a capacity of 12,280 grains per cubic foot; or 12.8 kgr/cu ft.

The performance of the resin during exhaustion is summarized in the attached Table 4. The first exhaustion produced a capacity of 12.8 kilograins per cubic foot to the alkalinity - chloride breakthrough. The next 11 cycles produced results which were too variable to consider. Regeneration to the free base form for these 11 cycles used dosages of ammonium hydroxide calculated to 125% of stoichiometric to the capacity obtained in exhaustion. Because of the poor performance, runs 12, 13, and 14 were made with the resin regenerated with ammonium hydroxide equivalent to 100% of the total capacity of the resin, which is several times the equivalent capacity realized. A stable average capacity of 10.8 kgr was obtained per cubic foot of resin, employing the excessive ammonium hydroxide regeneration.

Table 5 shows the typical partial analyses of effluent water samples collected from the exhaustion runs. The influent samples were a composite (drip sample) collected throughout the exhaustion; while effluent samples were collected at the end of the exhaustion to show ionic breakthrough. The partial analyses are presented to show the effect of the bicarbonate exchange process on the three ions, as well as to show the chloride breakthrough.

TABLE 4. ANION PERFORMANCE RECAP SHEET, per cubic foot. Regenerated at 1/2 gpm/cu ft (4 bed volume/hour), exhausted at 2 gpm/cu ft (16 bed volume/hour). Bicarbonate form of IRA-68 system.

	Regeneration		Exhaustion **			
RUN NO.	NH_4OH Used Kgr*	CO_2 Used Lbs	Bed*** Volumes	Load mg/l*	Capacity Kgr*	COMMENTS
1	0	3	86	340	12.8	New resin. Carbonation was difficult unfamiliar.
2-11	5-10	1-2	--	--	5-8	Results too variable.
12	33.9	4	92	275	11.1	Co-current regeneration for 12, 13,
13	33.9	3¼	90	264	10.4	14. Average capacity 10.8 Kgr.
14	33.9	3½	96	252	10.6	
15	13.5	2	54	245	5.8	Co-current regeneration, NH_4OH used at
16	13.5	2¼	84	220	8.1	125% of average capacity for 12, 13, 14.
17	13.5	3½	62	236	6.4	Leakage for 15 is high. Capacities are unstable.
18	13.5	1-3/4	79	260	9.0	Countercurrent regeneration, NH_4OH
19	13.5	1-3/4	68	296	8.9	used at the same 125% of 12, 13, 14.
20	13.5	2	62	294	8.0	Leakage for 18 & 19 is high, but capacity is fairly stable at average 8.6 Kgr. Concern expressed about resin fouling. Treated with two bed volumes of 3% HCl.
21	13.5	3-3/4	40	216	3.8	Clarifier, recarbonation by-passed.
22	13.5	2¼	45	218	4.3	Countercurrent regeneration, Drastic capacity loss questions loss of resin. Physical inspection shows resin present in good condition.
23	35.0 NaOH	1¼	34	214	3.2	Low capacity!
24	102	1¼	46	208	4.2	Regenerated high to determine maximum capacity obtainable.

* As $CaCO_3$ equivalent. ** to 10% Chloride break-thru. *** Volume resin occupies; 2 cu ft, or 15 gal. per bed volume.

TABLE 5. Anion resin (IRA-68) water sample analyses' abstracts collected during exhaustion. Results expressed in mg/l, as calcium carbonate equivalents. Bicarbonate form resin system.

Run No.	13	17	20	22	23	24
Alkalinity:						
Influent	150	102	78	404	330	326
Effluent	368	328	390	564	444	416
Chlorides:						
Influent	172	120	152	130	142	--
Effluent	40	60	20	34	52	58
Sulfate:						
Influent	97	116	144	88	100	--
Effluent	0	1	0	2	0	0

Note: Influent samples were composited throughout the exhaustion, effluent samples were spot-collected at the endpoint of the exhaustion.

An attempt was made to reduce the amount of regenerant required by returning the dosage to 125% of the stable average 10.8 kilograin capacity obtained in runs 12 - 14. This was a regenerant dosage of 13.5 kilograins (as calcium carbonate equivalent) for runs 15, 16, and 17. Co-current flows (downflow regeneration - downflow service) were used. The capacities obtained from these three runs were low and unstable, while the anion concentration to service was high.

In an attempt to improve the performance, counter-current procedures (downflow exhaustion - upflow regeneration) were adopted for runs 18, 19, and 20. The same regeneration level, 13.5 kilograins per cubic foot, was used. It produced a higher, fairly stable average capacity of 8.6 kilograins per cubic foot; however, anion leakage was of questionable quality.

At this point, concern was expressed about possible fouling of the resin. Therefore, the resin was washed with 2 bed volumes of 4% hydrochloric acid, downflow. The effluent during this acid treatment was sampled and analyzed for suspected contaminants, including sulfate, phosphate, iron, total organic carbon. No indication of foulants was found in the analyses. Table 6 shows the analyses of selected samples of the wash effluent where ionic concentrations peaked. The table also shows similar effluent analyses for samples collected during a later conducted sodium hydroxide wash. This latter wash is discussed below.

TABLE 6. Anion resin (IRA-68) wash sample analyses. Collected during clean-up procedures after separate exhaustions. All results in mg/l. Bicarbonate form resin system.

	Hydrochloric Acid Wash*			Sodium Hydroxide Wash*		
Bed volume thru	1.8	2.5	3.2	1.8	2.5	3.2
Sulfate, $CaCO_3$	2600	220	8	2600	1800	600
Phosphate, PO_4^{---}	28.5	9.4	1.5	340	183	145
Calcium, $CaCO_3$	15	6	3	2	0.4	0
Iron, Fe	36.6	4.8	0.6	0.8	1.3	0.8
Aluminum, Al	150	40	10	6.0	6.5	4.5
Organic Carbon, C	16	8	4	4625	2000	1750

* Two bed volumes of 4% strength, downflow at 0.5 gpm/cu ft.

It was postulated that high pH in the influent to the anion resin could be a cause for the irregular results. Therefore, the operation of the lime clarifier and recarbonation was discontinued for the next four runs. Pretreatment merely consisted of filtration through the dual media filter and activated carbon. Runs 21 and 22 were duplicate attempts made with counter-current regeneration using the 13.5 kilograins regeneration level. The average capacity obtained for the two exhaustions was 4.0 kilograins per cubic foot. The resin column was then opened for internal inspection, which showed no physical damage to the internals or loss of resin. A representative sample of the resin in the exhausted state was removed for laboratory analysis by Culligan and the resin manufacturer.

Resin analyses from both laboratories showed that the resin still possessed good chemical and physical properties. In addition, the resin manufacturer ran additional laboratory evaluation tests on the resin and concluded that the resin had been apparently properly regenerated and carbonated by our procedures.

Run #23 employed sodium hydroxide (NaOH) in an attempt to achieve better capacity. The sodium hydroxide is a stronger base than is the ammonium hydroxide previously used for regeneration. It should regenerate the resin more efficiently, and also better elute organics from the resin. Samples of the regenerant effluent were collected during the regeneration with sodium hydroxide. Table 6 shows the analyses of effluent samples. These samples did, indeed, show high concentrations of total organic carbon due to eluted organic material. The total organic carbon concentration in the regenerant effluent samples peaked at a value greater than 4,000 mg/l as carbon. This high organic level indicates that previous regenerations with ammonium hydroxide were insufficient to remove most of the organics from the resin. In spite of this organic elution and

high regenerant dosage, a low capacity (3.2 kilograins per cubic foot) was obtained.

A final attempt was made using a massive regeneration dosage (102 kilograins as calcium carbonate equivalent) of ammonium hydroxide. The low capacity (4.2 kilograins per cubic foot) obtained confirmed the agreement between the Project Officer of the Water Quality Office and the Project Manager at Culligan that the process was not suitable for demineralization of the secondary sewage effluent at Elgin, Ill.

The recommendation is therefore made that this process should not be applied to wastewater of salinities less than 600 mg/l. The following observations were offered as a basis for discontinuing studies on this process.

A. Operating procedures had not been fully established.

B. Recarbonation procedures, in particular, had not been detailed.

C. An unacceptably low volume of product water is produced to a typical operating endpoint.

D. Excessive anionic concentration in the product water occurs when the resin is exhausted to a high operating capacity.

E. High regenerant dosages, particularly for ammonium hydroxide, are necessary to obtain realistic capacities.

F. The resin manufacturer's published data indicates that the capacity of the resin decreases very rapidly as the influent salt content decreases below 500 mg/l, which is the approximate salt concentration of our influent water.

SECTION 8

STRONG ACID ION EXCHANGE RESIN PERFORMANCE-HYDROGEN FORM

This process employs a strong acid cation exchange resin. In the hydrogen form, this resin is capable of "splitting" ionized salts in water to remove cations by replacing them with an equivalent hydrogen ion. This resin has a cation - hydrogen selectivity such that neutral mineral salts in water are converted to their corresponding mineral acids. For example, the chlorides and sulfates of sodium, calcium, and magnesium are converted to the corresponding acids, as illustrated by the equation employing sodium chloride:

$$R\text{-}H + NaCl = R\text{-}Na + HCl$$

The effluent water, which has been treated by the hydrogen form resin, can then be treated with a bed of anion exchange resin (weak base) in the free base form. The function of this second bed of resin is to adsorb the acids produced by the cation exchanger. This is illustrated by the following equation employing the hydrochloric acid produced above:

$$RNH_2 + HCl = (R\text{-}NH_2)HCl$$

A combination of the two ion exchange beds will remove the mineral salts from the water. Incorporation of such a two bed ion exchange system after an efficient process for removal of organic contaminants will result in a reclaimed water without pollution due to inorganic and organic materials. This section of the report, however, discusses only the performance of the hydrogen form resin.

Our attempts to apply the hydrogen form of the strong acid cation exchange resin have been successful. We have used accepted techniques for the ion exchange industry in applying this resin to sewage treated by the activated sludge process and our pretreatment system. It is demonstrated, therefore, that the process can be used in the design of treatment plants.

The strong acid cation exchange resin was studied for treating the effluent from the pretreatment system, (settling, filtration, activated carbon adsorption). The clarifier and recarbonation components of pretreatment were not used. These latter components will remove phosphate, but this can be done by the anion exchange process as well. Turbidity removal will be accomplished easier by mechanical filtration than with clarification. Cost estimates based on influent water composition and the resin manufacturer's procedures for performance estimation show lime clarification to be costly. Table 7, "Comparison of estimated performances of strong acid cation exchange resin", details

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Table 7. Comparison of estimated performances of strong acid cation exchange resin (IRC-120). Resins treating activated sludge treated sewage, with and without lime clarification.

	With Lime	Without Lime
Composition of resin influent:		
Sodium, mg/l $CaCO_3$	298	205
Total hardness, mg/l $CaCO_3$	132	248
Chloride and Sulfate, mg/l $CaCO_3$	301	222
Alkalinity, mg/l $CaCO_3$	129	331
Total Cations, mg/l $CaCO_3$	430	553
Percent ratio of:		
Sodium	69	37
Calcium	15	28
Alkalinity	30	60
Performance prediction:		
Iso - capacity, Kgr/cu ft	10.4	10.3
Correction for alkalinity	1.06	1.12
Adjusted gross capacity, Kgr/cu ft	11.0	11.5
Deduct for rinse requirement, Kgr/cu ft	1.2	1.3
Net predicted capacity, Kgr/cu ft	9.8	10.2
Leakage, % of influent cations	12.0	4.0
Requirements for 1000 gallons:		
Resin, cu ft	2.57	2.83
Acid (66° Be sulfuric):		
pounds (3/cu ft)	7.7	8.5
Cost (1.8 ¢/lb), ¢	13.9	15.3
Lime:		
Pounds	4.6	0
Cost (1.0 ¢/lb), ¢	4.6	0
Chemical Cost (excluding recarbonation) total	18.5	15.3

the cost estimate for producing 1000 gallons of resin treated water with and without lime clarification. As shown, lime clarification increases the operating chemical costs from 15.3 to 18.5 cents per 1000 gallons of water from the cation resin. This cost does not include costs due to recarbonation which would apply only to the clarifier operation, although these costs should be a comparatively small factor.

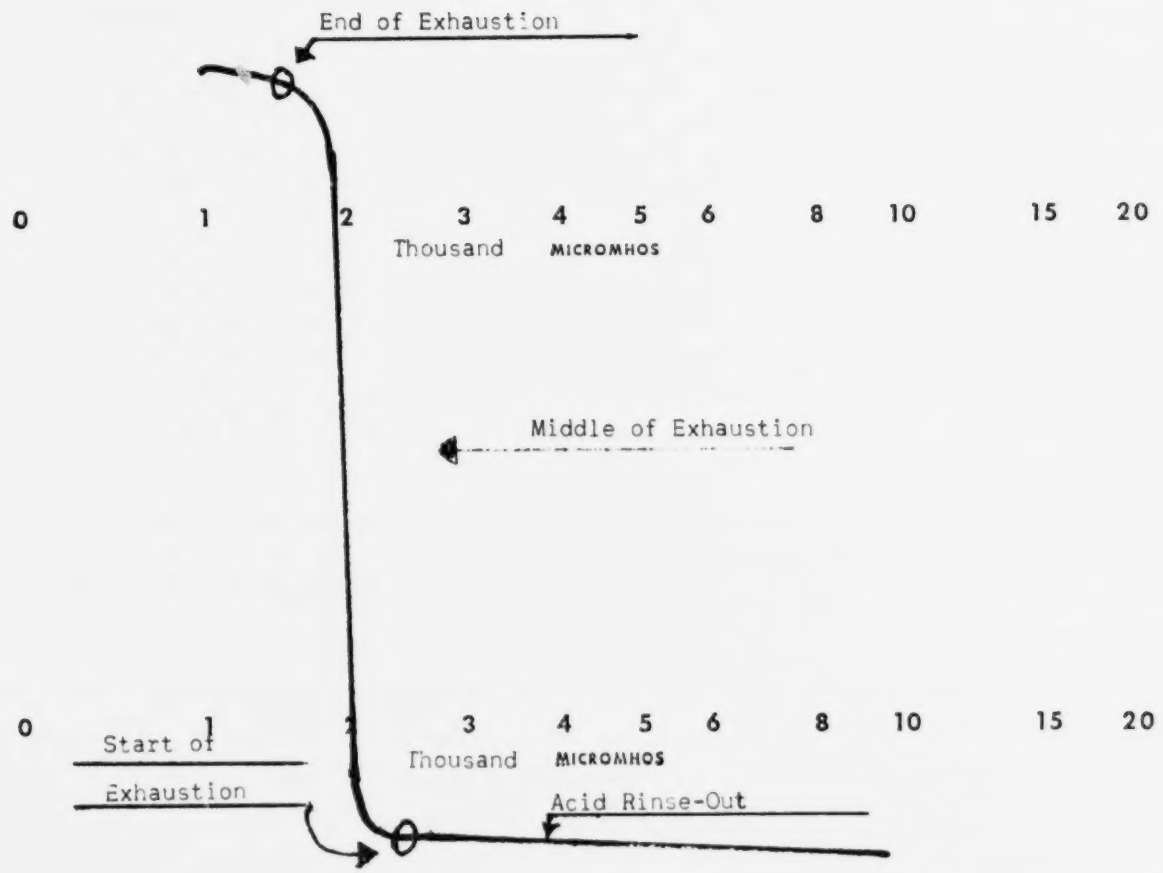
Studies of this strong acid cation exchange resin used 2 cu ft of resin in a 12" diameter tank, so that the height of resin was about 30" in place. Regeneration of the resin was accomplished with sulfuric acid, ranging from 2% to 5%. The use of sulfuric acid for regeneration can cause resin fouling by precipitation of calcium sulfate during regeneration. This precipitation in the resin is avoided by using an acid strength no greater than 2% when large amounts of calcium are being eluted. Increased capacity and reduced cation leakage to service can be obtained by increasing the strength and dosage of acid after eluting most of the calcium with an initial dosage of 2 pounds of acid per cubic foot of resin. This is sufficient to reduce the amount of calcium remaining on the resin so that the calcium sulfate solubility will not be exceeded when the stronger sulfuric acid solution is used. This procedure is refered to as "step-wise regeneration". Therefore, during regeneration, the first 2 pounds of sulfuric acid regenerant were prepared as a 2% solution while the remaining regenerant dosage beyond 2 pounds per cubic foot was fed at higher concentrations. We used regenerant dosages of 1 - 5 pounds of 66° Baume sulfuric acid per cubic foot of resin. Soft potable water was used for regenerant solution preparation and for the first portion of rinse. Final rinse was made with sewage water from the pretreatment system.

Performance of ion exchange resins during exhaustion was monitored by indicating and recording instruments. Figure 12, "Typical Effluent Conductivity From Cation (IRC-120) Resin", is a representative exhaustion effluent conductivity pattern, which shows three distinct zones as follows:

> High, but rapidly decreasing conductivity during regenerant acid rinse out.
>
> Moderate and stable conductivity during exhaustion. The conductivity is caused by the free mineral acidity formed by the hydrogen ion exchange for the cations.
>
> Rapidly decreasing conductivity, signaling increasing breakthrough of cations as the ion exchanger becomes exhausted.

The exhaustion cycles for the cation exchange resin were based on actual curves of this nature which were obtained for each run. The exhaustion started and ended when the effluent conductivity was 500 micromhos higher

Figure 12. Typical Effluent Conductivity From Cation (IRC-120) Resin


End of Exhaustion
0 1 2 3 4 5 6 8 10 15 20
Thousand MICROMHOS
Middle of Exhaustion
0 1 2 3 4 5 6 8 10 15 20
Thousand MICROMHOS
Start of Exhaustion
Acid Rinse-Out

and lower respectively than the average conductivity during service.

The total cationic loading to the cation exchanger was immediately determined by field analysis of a representative sample of the average influent water to the resin. This sample was collected at a steady, slow rate (drip sample). Two determinations were made on this sample. The first determination was for total alkalinity as collected (but after laboratory filtration). This determination equaled the total cations associated with alkalinity. The remaining cations were determined by passing the laboratory filtered sample through an analytical column packed with hydrogen form strong acid cation resin which was totally regenerated. The effluent from this laboratory column was analyzed for free mineral acidity (FMA), equal to the total cations except those associated with alkalinity. The latter cations are neutralized by the alkalinity present, and are therefore not determined in the FMA titration. The sum of these two results then equaled the total cation load in service to the cation exchange resin bed. The analytical laboratory at Culligan International Company was used to corroborate the field analyses as well as to obtain analyses in greater detail.

Table 8, "Strong Acid Cation Exchange Resin (IRC-120) Performance Recap", summarizes the performance of this resin. The top half of the table pertains to counter-current flows, while the bottom half pertains to co-current operation. A comparison of these two areas shows that the co-current flow gave better performance than did the counter-current. More capacity, less cation leakage in service, and better acid utilization resulted. Figures 13 and 14 attached are graphic representations of the regeneration efficiency.

Table 8 also shows that there is slightly better co-current performance at 3 gpm/cu ft than at 6 gpm/cu ft. Equipment designs can use the lower flow rate with the slightly better performance while considering the higher flow rate as a reserve for future expansion.

The requirements (of resin, regenerant acid, and rinse) to produce 1000 gallons of treated water are also shown on Table 8. Low regeneration levels decrease the operating costs (less pounds of regenerant) but increase the capital cost (more cubic feet of resin). Amortization favors use of low regenerant levels.

Tables 9, 10, and 11 attached, recap the individual exhaustion runs used in preparing the summary of Table 8. The data has been calculated to a "per cubic foot" base although more resin was actually used. The second column of these tables shows the weight of 66° Baume sulfuric acid used for regeneration. The third column shows the exhaustion flow rate in gallons per minute, which ranged from 3 - 6 gpm/cu ft.

The mineral loading during exhaustion is also shown in tables 9, 10, and 11.

Table 8. Strong acid cation exchange resin (IRC-120) performance summary. Per cubic foot base.

Co-current flows: downflow regeneration, downflow exhaustion.

Test Series	A	B	C	D	E	F
Regenerant, lbs/cu ft	5	5	3	3	1	1
Exhaust, gpm/cu ft	3	6	6	3	3	6
Capacity, kgr/cu ft	14.7	14.6	10.4	10.6	6.0	5.0
Acid Utilization %	44.0	43.7	52.0	53.0	90.0	74.9
Leakage, Average:						
mg/l	16.2	20.7	32.0	61.6	94.3	96.5
% of influent	2.7	3.6	5.6	10.4	16.4	17.6
Requirements per 1000 Gallons of water produced:						
Cation resin, cu ft	2.38	2.31	3.22	3.13	5.56	6.25
Acid, lbs 66° Be	11.9	11.6	9.7	9.4	5.6	6.3
Rinse water, at 35 gal/cu ft, gals.	83.3	80.9	113	110	195	219

Counter-current flows: upflow regeneration, downflow exhaustion.

Test Series	G	H	I	J	K
Regenerant lbs/cu ft	1	3	3	5	5
Exhaust, gpm/cu ft	3	3	6	6	3
Capacity, kgr/cu ft	4.7	8.0	8.1	10.1	10.2
Acid Utilization %	70.5	40.0	40.5	30.3	30.6
Leakage, Average:					
mg/l	103.3	63.7	64.7	68.1	64.8
% of influent	18.3	12.8	11.5	11.2	11.0
Requirements per 1000 Gallons of water produced:					
Cation resin, cu ft	7.04	3.64	4.00	3.51	3.38
Acid, lbs 66° Be	7.04	10.92	12.00	17.55	16.90
Rinse Water, at 35 gal/cu ft, gals.	246	127	140	123	118

Figure 13. CATION RESIN (IR-120) REGENERATION EFFICIENCY
Exhaustion Flow Rate, 6 gpm/cu ft

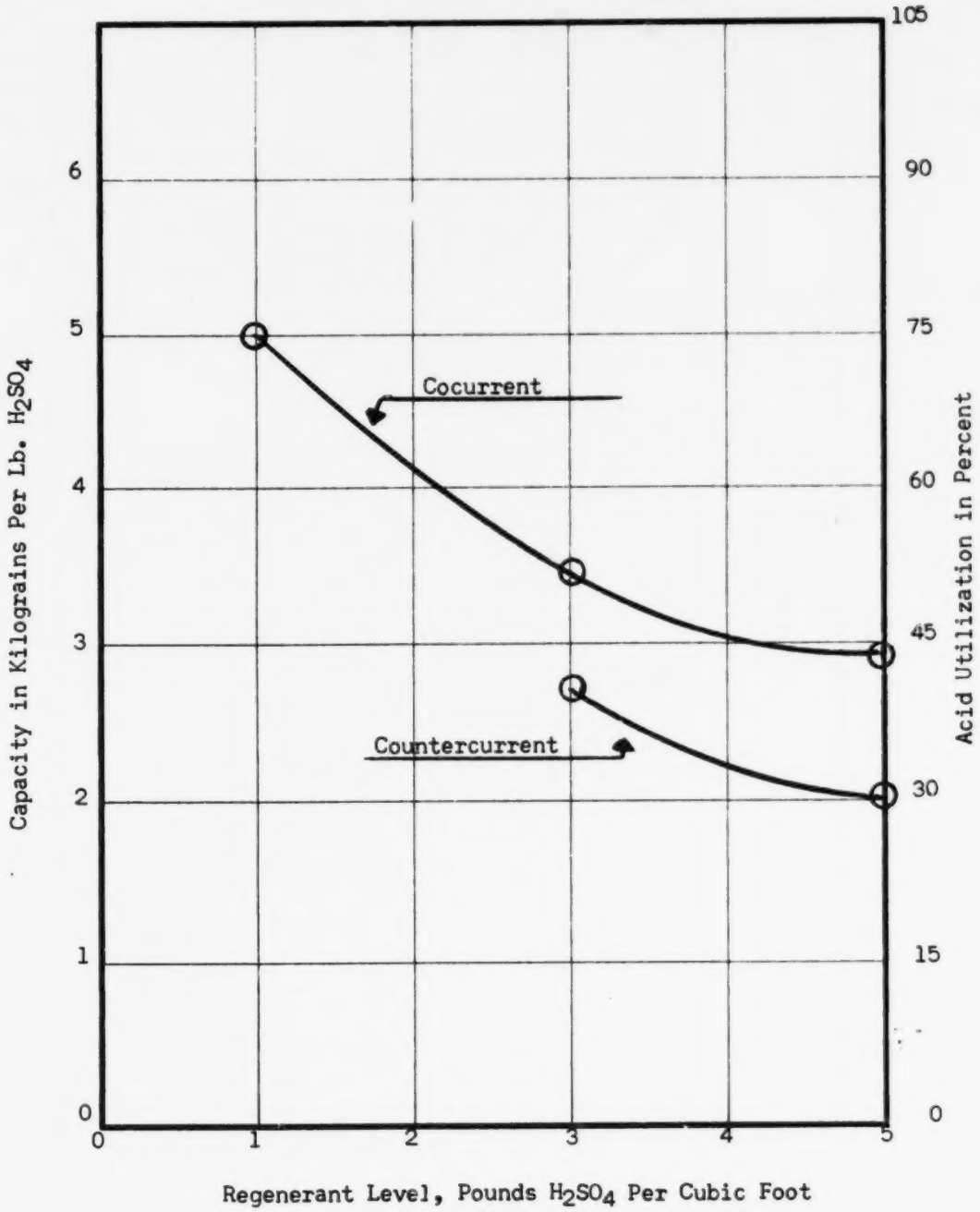


Figure 14. CATION RESIN (IR-120) REGENERATION EFFICIENCY
Exhaustion flow rate, 3 gpm/cu ft

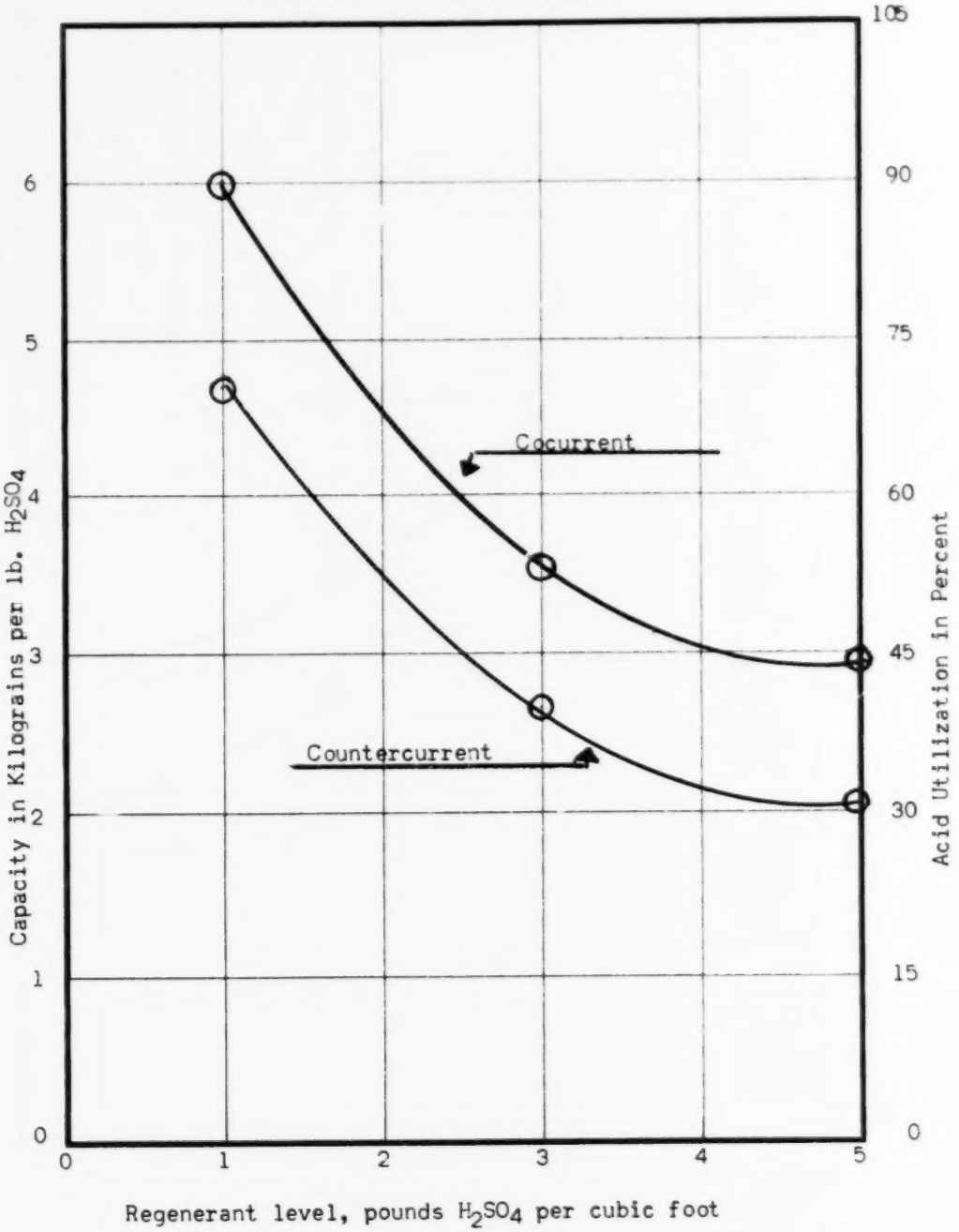


Table 9. CATION RESIN (IR-120) PERFORMANCE
RECAP SHEET

(per cubic foot)

Counter-current flows

Run #	H_2SO_4 lbs.	Exhaust GPM	Loading mg/l**	Capacity gals.	Kgr**
1	30	3	588	1070	36.8
2	5	3	592	523	18.1
3	"	"	552	436	14.1
4	"	"	566	350	11.6
5	"	"	592*	430*	14.9*
6	"	"	600*	425*	14.9*
7	"	"	600*	404*	14.2*
Series A		Average	597	420	14.7 (*based)
8	5	6	568	449	14.9
9	"	"	576	413	13.9
10	"	"	596	263	9.2
11	"	"	576	365	12.4
12	"	"	564*	435*	14.4*
13	"	"	584*	425*	14.5*
14	"	"	580*	435*	14.8*
Series B		Average	576	432	14.6 (*based)
15	3	6	592	319	11.0
16	"	"	592*	307*	10.6*
17	"	"	592*	296*	10.2*
18	"	"	592*	300*	10.4*
19	"	"	572*	312*	10.4*
20	"	"	556*	329*	10.7*
21	"	"	550*	325*	10.5*
Series C		Average	576	311	10.4 (*based)

** As calcium carbonate equivalent.

Table 10. CATION RESIN (IR-120) PERFORMANCE RECAP SHEET

(Per Cubic Foot)

Counter-current flows

Run #	H_2SO_4 lbs	Exhaust GPM	Loading mg/l**	Capacity gal.	Capacity Kgr**
22	3	3	572	348	11.6
23	"	"	584	326	11.1
24	"	"	588	308	10.5
25	"	"	608	307	10.9
26	"	"	584	224	7.6
27	"	"	568*	322*	10.7*
28	"	"	568*	319*	10.6*
29	"	"	592*	315*	10.8*
30	"	"	564*	319*	10.5*
Series D Average			573	319	10.6 (*Based)
31	1	3	524*	203*	6.1*
32	"	"	572*	179*	6.0*
33	"	"	600*	171*	6.0*
34	"	"	600*	168*	5.9*
35	"	"	580*	178*	6.0*
Series E Average			575	180	6.0 (Based)
36	1	6	528*	168*	5.0*
37	"	"	532*	167*	5.2*
38	"	"	548*	153*	4.9*
39	"	"	580*	150*	5.1*
Series F Average			547	160	5.0 (Based)

** As calcium carbonate equivalent.

Table 11. CATION RESIN (IR 120) PERFORMANCE
RECAP SHEET

(Per Cubic Foot)
Co-current Flows

Run #	H_2SO_4 lbs	Exhaust gpm	Loading mg/l**	Capacity gal.	Kgr.**
40-44	1(upflow)	3	Conditioning runs on resin transferred to smaller tank for upflow studies.		
45	"	"	562*	133*	4.4*
46	"	"	556*	160*	5.2*
47	"	"	Invalid results due to mechanical		
48	"	"	difficulties		
49	"	"	576*	133*	4.5*
Series G Average			565	142	4.7 (*Based)
50	3(upflow)	3	488	198	5.7
51	"	"	484*	291*	8.2*
52	"	"	490*	264*	7.6*
53	"	"	516*	271*	8.1*
Series H Average			497	275	8.0 (*Based)
54	3(upflow)	6	546	261	8.3
55	"	"	564*	240*	7.9*
56	"	"	464*	301*	8.2*
57	"	"	596	246*	8.6*
58	"	"	592*	233*	8.1*
59	"	"	598*	229*	7.9*
Series I Average			561	250	8.1 (*Based)
60	5(upflow)	6	608*	293*	10.4*
61	"	"	608*	260*	9.2*
62	"	"	612*	285*	10.2*
63	"	"	608*	300*	10.6*
Series J Average			609	284	10.1 (*Based)
64	5(upflow)	3	616	301	10.9
65	"	"	584*	297*	10.5*
66	"	"	588*	291*	10.0*
67	"	"	592*	291*	10.1*
Series K Average			588	297	10.2 (*Based)

** As calcium carbonate equivalent.

The last two columes of the three tables show the capacity obtained during exhaustion. The gallons shown are those number of gallons delivered between the two selected start and endpoint conductivity values for the effluent as previously discussed.

The capacity has been calculated from the gallons produced and the loading factor to obtain the values shown as kilograins (kgr). The loading factor is shown as mg/l, as calcium carbonate equivalent: this is changed to grains per gallon (gpg) by dividing by 17.1. The capacity, in kgr/cu ft is then calculated from the following formula:

$$\text{kgr/cu ft} = \frac{VL}{17.1 \times 1000}$$

Where: kgr/cu ft is the ion exchange capacity per cubic foot of resin.

V is the number of gallons treated by <u>one</u> cubic foot of resin.

L is the loading factor, in mg/l as calcium carbonate equivalent.

The performance obtained with this resin is encouraging. The results do not differ greatly from those which would be predicted on a potable water. The published procedures of Rohm & Haas were used to predict the capacity and leakage from this cation exchange resin on potable water. The capacity and leakage predicted by calculation, as compared with the values obtained for the first four test series, are shown on the attached Table 12, "Cation Resin (IRC-120) Performance, Comparison of Estimates and Actual". This comparison shows that the predicted capacities are comparable to those which are actually obtained. Cation leakage from the exchanger, however, has been somewhat higher than predicted values.

The quality of water produced from the cation exchange resin during service is shown in the attached Table 13. Each test series consisted of several regeneration and exhaustion cycles made until stable performance was obtained. Table 13 contains typical results of the analyses made of composite samples of influent and effluent during the stable performance runs. As shown, the influent contains a typical turbidity of 2.0 - 3.4 JTU; the mechanical filtration of the pretreatment system was performing adequately. The table also shows that the influent total organic carbon ranged from 5.4 - 10.2 mg/l. (The previous section of this report which discussed the pretreatment equipment stated that the activated carbon filter of the pretreatment system removed about one-half of this organic carbon from the sewage). This Table 13 shows that the total organic carbon was unaffected by the cation exchange resin.

Table 12. Cation Resin (IR-120) Performance Comparison - Estimates and Actual

Test Series	A	B	C	D
Regenerant, lbs/cu ft	5	5	3	3
Exhaust, gpm/cu ft	3	6	6	3
Loading, mg/l as $CaCO_3$:				
Ca	166	144	156	145
Mg	188	198	202	187
Monovalents	246	234	214	260
Alkalinity	342	312	344	374
Loading %:				
Ca	28	25	27	25
Mg	31	35	35	31
Monovalent	41	40	38	44
Alkalinity	57	54	60	63
Capacity, kgr/cu ft:				
Predicted:				
Unadjusted	14.4	14.6	10.4	10.4
Adjusted	1.12	1.12	1.13	1.13
Adjusted Gross	16.1	16.4	11.8	11.8
Rinse Deduct	1.3	1.2	1.2	1.2
Net Prediction	14.8	15.2	10.6	10.6
Obtained	14.7	14.6	10.4	10.6
Leakage %:				
Predicted	2	2	4	4
Obtained	2.7	3.6	5.6	10.4

Table 13. Water analyses summary of typical exhaustion of cation exchange resin (IRC-120)

Test Series	A	B	C	D	E	F
Regenerant, lb/cu ft	5	5	3	3	1	1
Exhaust, gpm/cu ft	3	6	6	3	3	6
Influent:						
pH, units	7.7	8.0	8.0	8.1	7.8	8.0
Turbidity, JTU	3.4	2.4	2.0	3.0	2.6	2.8
Total organic carbon mg/l C	5.4	5.5	7.2	8.6	10.2	9.9
Magnesium, mg/l $CaCO_3$	192	193	195	193	177	174
Calcium, mg/l $CaCO_3$	164	159	169	155	146	161
Sodium, mg/l $CaCO_3$	207	178	163	164	223	175
Potassium, mg/l $CaCO_3$	10.0	11.4	11.0	11.2	11.7	10.5
Ammonia, mg/l N	2.0	2.6	2.0	6.7	3.3	5.8
Effluent:						
Total organic carbon mg/l C	5.3	5.4	6.6	9.5	9.0	4.6
Acidity, mg/l $CaCO_3$	217	206	191	172	133	144
Magnesium, mg/l $CaCO_3$	Trace	1.7	0.9	9.4	4.3	6.0
Calcium, mg/l $CaCO_3$	Trace	5.3	8.2	8.4	6.8	10.0
Sodium, mg/l $CaCO_3$	12.8	12.8	19.7	25.8	71.1	47.9
Potassium, mg/l $CaCO_3$	1.8	1.6	1.9	2.3	4.4	4.6
Ammonia, mg/l N	0.0	0.0	0.0	1.3	2.7	2.9

The effluent analyses also show the production of acidity (which would result in a low pH) due to the hydrogen ion exchange for cations. This acidity can be easily removed by subsequent ion exchange treatment with a second ion exchange resin as discussed in later sections of this report. The analyses show great reduction in the cationic composition of the influent water. Ammonia is significantly reduced; however, total avoidance of this cation in the effluent water will require subsequent break-point chlorination techniques.

Regeneration of this cation exchange resin with sulfuric acid has been accomplished without difficulty. Precipitation of calcium sulfate in the resin bed has been avoided; however, such precipitation occurs in the regeneration effluent about 10 minutes after leaving the column. The composition of regeneration effluents, as shown in Table 14, duplicate those from conventional procedures. Specific analyses for total organic carbon elution show only analytical and sampling variations throughout the regeneration. Excess acid, which requires disposal, is present in the regeneration effluent. Neutralization of this acid is discussed in a later separate section of this report. An alternate means of acid disposal is by its reuse for the regeneration of a weak acid cation exchange resin, which is a separate ion exchange system, also discussed in a later section.

The recommendation is made that strong acid cation exchange resin can be applied to treat the effluent from secondary sewage treatment plants. Use of the strong acid cation exchange resin, of course, is only a partial means of wastewater demineralization. The effluent is unacceptable and must be post treated. Subsequent sections in this report will consider the application of ion exchange treatment for this post treatment to produce a demineralized wastewater with acceptable mineral concentration. The operating material requirements for resins, regenerants, rinse water, etc. is developed for each total ion exchange system in portions of the section titled, "Operating Material Requirements for Ion Exchange Processes".

Table 14. Regeneration effluent analyses from strong acid cation resin IRC-120.

WATER ANALYSIS

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TEST (*AS $CaCO_3$)									
pH									
ACIDITY, MINERAL *	1000	5300	6600	7600	8100	3100			
ALKALINITY, OH*									
ALKALINITY, CO_3*									
ALKALINITY, HCO_3*									
CHLORIDES*	40	38	40	40	38	40			
TURBIDITY IN J.T.U.									
SULFATES*									
TOTAL PHOSPHATE AS PO_4									
TOTAL HARDNESS*	7200	7700	6300	2900	2100	340			
MAGNESIUM*	5400	4400	3400	1700	1300	200			
CALCIUM*	1800	3300	2900	1100	800	140			
AMMONIA NITROGEN AS N	10	140	220	230	110	40			
NITRATE NITROGEN AS N									
NITRITE NITROGEN AS N									
SODIUM*	5100	3000	2600	1800	1600	260			
POTASSIUM*	250	100	100	120	100	15			
TOTAL ORGANIC CARBON	10.5	11.0	10.1	9.8	10.6	9.3			
TOTAL DISSOLVED SOLIDS									
Bed Volume	1	1-2/3	2	2-1/3	2-2/3	3			

Regeneration Effluent IRC-120

SECTION 9

WEAK BASE ANION EXCHANGE RESIN PERFORMANCE-FREE BASE FORM

This process employs a weak base anion exchange resin. In the free base form, this resin is capable of adsorbing strong acids. In multi-column demineralization processes, such acids are produced by the strong acid cation exchange resin.

Whereas the strong acid exchange resin removes cations to form both strong acids and weak acids, the weak base anion exchange resin can only adsorb strong acids. Weak acids are not removed by the weak base anion exchange resin. One weak acid not removed is carbonic acid, produced by the cation exchange of an alkaline salt, as illustrated by the following equation employing sodium bicarbonate:

$$RH + NaHCO_3 = RNa + H_2CO_3$$

$$H_2CO_3 = H_2O + CO_2$$

The carbon dioxide produced is unaffected by the weak base anion exchange resin: the gas is readily eliminated through established technology with aeration or degasification.

The performance characteristics of the weak base anion exchange resi have been encouraging. The technology for their application is sufficiently developed for application to wastewater demineralizatio Standard techniques of the ion exchange industry for application of this resin have been employed. This resin successfully treated the effluent from a strong acid cation exchange resin (hydrogen form). It is demonstrated, therefore, that a combination of the two resins can be used in the design of equipment for demineralization of secondary sewage effluents.

The product water produced by the above system is easily degased to remove carbon dioxide. The resulting product will have a low conter of dissolved solids. Total phosphate will be reduced to a typical average concentration of about one mg/l; further reduction is possil by treating less water per cycle with a corresponding increase in chemical operating costs per unit volume of water produced.

Ammonia leakage to service does occur from the cation exchange resi and this ammonia is unaffected by anion exchange. The ammonia can l eliminated from the product water by break-point chlorination. Nit nitrogen was a constant 0.1 mg/l as nitrogen in the effluent. Nitr nitrogen was present to a much lesser degree.

Algae regrowth did not occur during an approximate one month's storage in vented clear glass bottles which were half filled. Product water was collected directly from the anion exchanger three times during the exhaustion cycle; these were at the start, middle, and at the end of exhaustion. These samples were exposed to direct sunlight on the south-end of the pilot plant during early fall. All samples remain clear, with no noticeable growth appearing.

Degasification of the product water was also studied. Product water from the weak base anion exchange resin contains dissolved carbon dioxide equivalent to the bicarbonate alkalinity of the influent water. This carbon dioxide should be removed from the product water if this water is to be transported in pipes. Mechanical degasification was readily obtained without difficulty. Foaming in the degasifier was not observed; nor were growths or precipitate formation in the system observed.

The performance characteristics of two different weak base anion resins were studied. A macroporous resin (IRA-93) has been used. This resin has large pores, thereby minimizing the tendancy of organic fouling. Sewage effluent normally contains ionic organic chemicals of comparatively large size. The organic molecules can be partially removed by anion exchange resin; however, they usually are difficult to elute during regeneration. This difficulty is minimized with macroporous resin.

A second weak base resin (IRA-68) has been similarily evaluated. This resin also possesses a high degress of porosity. It has been used efficiently in industry as a decolorizing agent in a variety of situations. Because of its resistance to organic fouling, it was established that both types of weak base resin should be evaluated.

Both weak base anion exchange resins were individually studied in 10" diameter tanks containing 1-1/2 cu ft of resin. This amount of resin resulted in a bed height of about 31".

Regeneration was with a 4% solution of ammonium hydroxide made in soft potable water. This water was used to avoid precipitation of polyvalent cations in the regenerant solution. The solution was prepared in the open regenerant solution vessel, and pumped through the anion resin bed. Rinse was in two stages: first with approximately 2-1/3 bed volumes of softened water at the regeneration flow rate; second, with water from the cation exchange resin (as in service) at the service flow rate.

Observation of the recorded effluent conductivity indicated when the fast rinse was finished and the service exhaustion was initiated. Prior to this point, the conductivity decreased rapidly; during service it was stablized; at exhaustion, the conductivity increased rapidly. Capacity

was calculated based on a conductivity endpoint fifty micromhos higher than the stable minimum conductivity obtained during service.

Capacity calculations for the resin were based upon the above determined volume of water and the average loading factor of the water from the cation exchange resin during the exhaustion. The loading factor was determined from the analysis of the composite sample of the influent. The sample was titrated for its content of free mineral acidity (FMA). FMA is what must be removed by the weak base resin.

The performance of each of the two resins is discussed separately below.

Weak base resin IRA-93 performance.

The performance of the IRA-93 weak base anion exchange resin is summarized in the attached Table 15. As shown, capacities of about 17 kilograins per cubic foot were obtained under the conditions investigated. The capacity obtained is compatible with the resin manufacturer's (Rohm & Haas) literature, which shows 21.0 kilograins. The slight capacity difference is probably due to the effect of unknown organic loading. The only significant difference in capacity under varied operating conditions was obtained by reducing the exhaustion flow rate from 6 gpm to 2 gpm per cubic foot. This flow reduction increased the capacity obtained by about 10%. This capacity increase is incufficient to justify the increased plant size which would be necessary to accommodate the slower flow rate.

Table 15 shows the average leakage of phosphate to be about 1 mg/l. A lower level could be obtained by reducing the operating capacity. Table 16 shows the phosphate break-through from this weak base anion resin during a typical exhaustion cycle. As shown, after treating 1,380 gallons of water, the effluent phosphate concentration was 17.6 mg/l phosphate as PO_4^{---}. The average concentration in the effluent was 1.2 mg/l PO_4^{---}. Obviously, avoiding the higher phosphate concentration present at the end of the exhaustion cycle would reduce the average phosphate concentration in the treated water.

Individual results for the test on the IRA-93 resin are summarized in Table 17, "Anion Resin (IRA-93) Performance Recap Sheet". As shown, stable capacities were quickly obtained. The regenerant dosage used ranged from 75% to 117% stoichiometric to the maximum capacity of the resin.

The ionic concentration of the effluent from the IRA-93 resin (which has previously been treated with cation exchange resin in hydrogen form) is shown in Table 18 for selected samples during a typical exhaustion cycle. The table shows low concentrations for both cations and anions. Phosphates, however, increase rapidly as the endpoint is approached. As previously discussed, these higher concentrations can be easily avoided.

TABLE 15. Weak base anion exchange resin (IRA 93) performance summary. Treating effluent from cation exchange (per cubic foot)

Test Series	A.	B.	C.	D.	E.
Regeneration:					
NH_3 (28%) lbs	7.3	7.3	6.6	6.6	4.8
NH_4OH (100%) lbs	4.2	4.2	3.8	3.8	2.8
Direction	Down	Down	Down	Down	Down
Flow, gpm	0.3	0.3	0.3	0.9	0.9
Exhaustion:					
Direction	Down	Down	Down	Down	Down
Flow, gpm	6	2	6	6	6
Capacity, Kgr	17.1	18.8	16.9	17.4	17.0
Ammonia utilization, percent	42	46	46	47	62
Leakage, average mg/l PO_4^{---}	2.5	0.5	1.3	1.4	1.2
Requirements per 1000					
Gallons produced:					
Anion resin, cu ft	0.66	0.58	0.71	0.67	0.75
Ammonia, 28%, lbs	4.75	4.19	4.70	4.48	3.58
Rinse water, gal.	75	75	69	70	65

TABLE 16. Phosphate break-thru from weak base anion resin (IRA-93) during service exhaustion on cation resin effluent, which contains 19.2 mg/l phosphate. Per cubic foot basis.

Gallons to service	Phosphate, mg/l, PO_4^{---}	Effluent Conductivity micromhos
0	0.5	---
667	0.2	---
1270	1.5	---
1310	3.2	40
1330	6.4	50
1350	12.0	60
1360	10.4	70
1370	14.8	80
1380*	17.6	90
1385	17.6	100
Composite Average	1.2	---

* Endpoint of service exhaustion.

TABLE 17. ANION RESIN (IRA-93) PERFORMANCE
RECAP SHEET
Treating Cation Resin Effluent
(per cubic foot)

Run #	NH_4OH (100%) Lbs **	Exhaust GPM	Loading mg/l	Capacity gals.	Capacity gr
1	New	6	---	---	---
2	2.8	"	200	1270	14.8
3	"	"	220	1277	16.4
4	"	"	220	1193	15.3
5	4.2	6	176	1314	13.5
6	"	"	208	1247	15.2
7	"	"	incompleted run		
8	"	"	204	1361	16.2
9	"	"	212	1167	14.5
10	"	"	200	1227	14.4
11	"	"	134*	1584*	17.0*
12	"	"	200*	1454*	17.1*
13	"	"	192*	1524*	17.1*
Series A average (* based)			192	1521	17.1
14	4.2	2	172*	1761*	17.6*
15	"	"	200*	1700*	19.8*
16	"	"	192*	1684*	18.9*
Series B average (* based)			188	1715	18.8
17	3.8	6	112	2094	13.7
18	"	"	196*	1487*	17.0*
19	"	"	212*	1354*	16.8*
20	"	"	212*	1369*	17.0*
Series C average (* based)			207	1403	16.9
21	3.8	6	210	1383	17.0
22	"	"	incomplete run		
23	"	"	212	1253	15.5
24	"	"	216	1384	17.5
25	"	"	220	1035	13.3
26	"	"	*204	*1421	*16.9
27	"	"	*200	*1525	*17.8
Series D average (* based)			202	1473	17.4
28	2.7	6	*216	*1360	*17.2
29	"	"	*220	*1367	*17.2
30	"	"	*220	*1301	*16.7
Series E average (* based)			219	1343	17.0

** 3.6 lbs is stoichiometric to maximum capacity of resin.

Table 18. Ionic break-thru from weak base anion resin (IRC-84) during service exhaustion.

WATER ANALYSIS

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TEST (*AS $CaCO_3$)	Raw	Carbon Filtered	Cation Effluent		Anion Effluent			
pH	8.0	8.1						
ACIDITY, MINERAL *			220.					
ALKALINITY, OH*								
ALKALINITY, CO_3*								
ALKALINITY, HCO_3*					12.	10.	2.	12.
CHLORIDES*			140.		6.	0.	4.	0.
TURBIDITY IN J.T.U.	8.0	1.8						
SULFATES*			56.		0.	0.	0.	0.
TOTAL PHOSPHATE AS PO_4	16.0	17.6	19.2		0.5	0.2	27.2	1.2
TOTAL HARDNESS*								
MAGNESIUM*								
CALCIUM*								
AMMONIA NITROGEN AS N								
NITRATE NITROGEN AS N			1.4		0.	0.	0.	0.
NITRITE NITROGEN AS N								
SODIUM*								
POTASSIUM*								
TOTAL ORGANIC CARBON	20.4	8.4	7.2		6.5	7.5	9.1	7.7
TOTAL DISSOLVED SOLIDS								
Number	956	955	953		950	951	952	954
Gallons For ½ cu ft	Drip	Drip	Drip		0	1000	2078	Drip

IRA 93 Run No. 24

WATER ANALYSIS

FWPCA Contract No. 14-12-599

DATE October, 1970

TEST (*AS $CaCO_3$)									
Micromhos	30	40	50	60	70	80	90	100	---
ACIDITY, MINERAL *									
ALKALINITY, OH*									
ALKALINITY, CO_3*									
ALKALINITY, HCO_3*	6.	6.	8.	6.	4.	6.	1.	2.	12
CHLORIDES*	0.	0.	0.	0.	0.	2.	2.	6.	0
TURBIDITY IN J.T.U.									
SULFATES*	0.	0.	0.	0.	0.	0.	0.	0.	0
TOTAL PHOSPHATE AS PO_4	1.5	3.2	6.4	12.0	10.4	14.8	17.6	17.6	1.2
TOTAL HARDNESS*	0.	0.	0.	0.	0.	0.	0.	0.	---
MAGNESIUM*									
CALCIUM*									
AMMONIA NITROGEN AS N	0.	0.	0.3	0.3	0.3	0.3	0.3	0.	---
NITRATE NITROGEN AS N	0.	0.	0.	0.	0.	0.	0.	0.	0
NITRITE NITROGEN AS N									
SODIUM*	6.8	7.3	7.3	7.5	7.3	7.9	8.2	7.8	---
POTASSIUM*	0.2	0.2	0.3	0.2	0.2	0.1	0.1	0.1	---
TOTAL ORGANIC CARBON	9.8	8.0	10.8	15.1	13.3	13.6	16.1	14.1	7.7
TOTAL DISSOLVED SOLIDS									
Number	957	958	959	960	961	962	963	964	954
Gallons For ½ cu ft	1908	1965	1998	2020	2038	2053	2065	2079	Drip

Break-thru samples, IRA-93, Run No. 24

The influent to this anion exchange system has been treated with activated carbon; consequently, the influent total organic carbon was typically 6 - 8 mg/l as carbon, and the composite (drip) shows the treated effluent to contain an average of 7.7 mg/l. However, the analyses of samples collected as exhaustion approach showed breakthrough of organic materials from this resin. Therefore, organics are removed by IRA-93 to a small extent.

The regeneration of the IRA-93 has been characterized when 4.2 lbs of ammonium hydroxide were used; this was equal to 115% of the stoichiometric requirement for the total capacity of the resin. The regenerant effluent was sampled in 1/4 or 1/2 bed volume increments. The analyses for the samples collected within the range of 1 to 3-3/4 bed volumes are shown in Table 19, "Regenerant Effluent Analyses From Weak Base Anion Resin (IRA-93)". As shown, regenerant ammonium hydroxide was initially neutralized by the adsorbed acids (chloride, sulfate, and phosphate) so that low alkalinity resulted. However, by the third bed volume, most of the ammonium hydroxide was not being used. The concentration of ammonium nitrogen shows that the regenerant concentration reached a maximum at about 2 bed volumes and started to decrease at around 3 bed volumes. Elution of chlorides is slower than for the other anions. However, this will not be of significance during the service cycle.

Total organic carbon elution from this resin is difficult, as seen by Table 19. The consequence of this is that organic fouling of the resin may result. This in turn, would result in reduced capacity and performance during service. Remedial maintenance procedures may be required to remove the organics. However, stable capacities were observed during the studies.

Pilot plant studies have shown that weak base resin IRA-93 is easily applied in demineralization of secondary sewage. Stable capacities were readily achieved; a reasonable quantity of water was treated; regeneration with ammonium hydroxide was without difficulty. Although the possibility of organic fouling of this resin cannot be ignored, it was not encountered. Excess ammonium hydroxide is required to regenerate the resin. This excess can be neutralized or recovered for reuse. Additional treatment beyond demineralization is recommended: first, degasification for removal of carbon dioxide; second, break-point chlorination for disinfection and removal of ammonium ion leakage.

Weak base resin IRA-68 performance.

The performance of the IRA-68 weak base anion exchange resin is summarized in Table 20. As shown, operating capacities of about 24 kilograins per cubic foot were obtained, representing 70% of the total capacity of the resin.

TABLE 19. Regenerant effluent analyses from weak base anion resin (IRA-93). Regenerated with 4.2 lbs of 100% ammonium hydroxide.

WATER ANALYSIS

FWPCA Contract No. 14-12-599 DATE September, 1970

TEST (*AS $CaCO_3$)										
pH										
ACIDITY, MINERAL *										
ALKALINITY, OH*						17500.	24800.	13900.	13300.	9600.
ALKALINITY, CO_3*						22000.	10400.	10200.	3600.	4000.
ALKALINITY, HCO_3*	60	580	762.	3200.	14700					
CHLORIDES*	40	880	---	2170.	26500.	1200.	800.	---	600.	580.
TURBIDITY IN J.T.U.										
SULFATES*	0	56	2250.	16800.	20000.	90.	33.	22.	11.	11.
TOTAL PHOSPHATE AS PO_4	3.9	1230	41400.	77000.	690.	8.8	3.0	2.0	1.3	3.0
TOTAL HARDNESS*										
MAGNESIUM*										
CALCIUM*										
AMMONIA NITROGEN AS N	30.0	553	5430	12180	14000	14000	12180	7660	5190	407
NITRATE NITROGEN AS N	0.3	515	35.0	107.5	107.5	10.0	9.0	8.7	8.2	7.6
NITRITE NITROGEN AS N	0.1	0.6	2.5	15.0	6.4	0.2	0.3	0.3	0.1	0.1
SODIUM*										
POTASSIUM*										
TOTAL ORGANIC CARBON	10.1	22.6	33.8	35.0	37.4	38.2	29.2	27.4	21.8	22.8
TOTAL DISSOLVED SOLIDS										
Number	860	861	862	863	864	865	866	867	868	869
Bed Volume	1	1/4	1/2	1-3/4	2-1/4	2-3/4	3	3 1/4	3 1/2	3-3/4

IRA 93 Regeneration Effluent

TABLE 20. Weak base anion exchange resin (IRA-68) performance summary. (per cubic foot basis). Treating cation resin effluent.

Test Series	A.	B.	C.	D.	E.	F.
Regeneration:						
NH_3 (28%) lbs	5.5	5.5	5.5	5.5	4.6	5.5
NH_4OH (100%) lbs	3.2	3.2	3.2	3.2	2.8	3.2
Direction	Down	Down	Down	Down	Down	Down
Flow, gpm	2/3	1/2	1/2	1/2	1/2	1/3
Exhaustion:						
Direction	Down	Down	Down	Down	Down	Down
Flow, gpm	6	4	2-1/3	6	6	6
Capacity, Kgr	21.7	24.8	24.4	24.5	23.6	25.0
Ammonia utilization percent	68	77	76	76	86	78
Leakage, average mg/l PO_4^{---}	0.2	1.5	0.4	0.8	1.2	0.6
Requirements per 1000 Gallons produced:						
Anion resin	0.54	0.53	0.53	0.55	0.66	0.55
Ammonia, 28% lbs	2.96	2.89	2.89	3.02	3.03	3.03
Rinse water, gal.	25	27	29	27	34	27

4% ammonium hydroxide was employed for downflow regeneration of the resin. In five of the six test series, the regenerant dosage has been 3.2 pounds of ammonium hydroxide per cubic foot - as specified by the resin manufacturer. In test series E, however, dosage was reduced to 2.8 pounds per cubic foot in an attempt to achieve better regenerant utilization. This lower regeneration level increased the ammonia utilization from about 75 to 86% of the applied dosage. The reduced dosage did not significantly affect capacity.

Further inspection of the summary Table 20 (test series A, B, and F) shows that regeneration flow rates of 1/2 and 1/3 gpm per cubic foot produced equivalent capacities (24.5 and 25.0 kilograins). Increasing the regeneration flow rate to 2/3 gpm per cubic foot, however, reduced the capacity to 21.7 kilograins. Varying the exhaustion flow rate between 2 and 6 gpm per cubic foot (test series B, C and D) did not affect the performance significantly.

The table also shows the average leakage of phosphate (as PO_4^{---}) is no greater than 1.5 mg/l. This phosphate concentration is similar to that obtained with IRA-93 weak base anion resin previously discussed. Also, a lower phosphate level can be obtained by reducing the operating capacity.

Table 21 shows the phosphate break-through from the IRA-68 resin during a typical service exhaustion. After treating 2,000 gallons of water, the effluent phosphate concentration was 30.0 mg/l phosphate as PO_4^{---}. The phosphate concentration in the composite effluent was 0.6 mg/l. Obviously, as in the case with the IRA-93 resin, avoiding the higher phosphate concentration present at the end of the exhaustion cycle would reduce the average phosphate concentration in the treated water.

Individual results for the tests on the IRA-68 resin have been summarized in Table 22, "Anion Resin (IRA-68) Performance Recap Sheet". This sheet shows that stable capacities were quickly obtained.

The ionic concentration of the effluent from the IRA-68 resin is shown in Table 23 for selected samples during a typical service exhaustion. This table is similar to Table 18 presented for IRA-93 resin. A comparison of these two tables shows that the IRA-93 resin has less chloride leakage than does the IRA-68. The phosphate concentration is also lower during break-through with the IRA-93. An important feature in the comparison is the concentration of total organic carbon: during exhaustion, IRA-93 shows (Table 18) some break-through of organic carbon, while IRA-68 does not. Organic compounds are apparently not accumulating on the resin. The performance of the resin in service does not indicate fouling tendencies.

TABLE 21. Phosphate break-thru from weak base anion resin (IRA-68) during exhaustion with cation effluent. Influent contains 20.8 mg/l phosphate. Per cubic foot basis.

Gallons to service	Phosphate, mg/l, PO_4^{---}	Effluent Conductivity micromhos
0	0.2	---
1000	0.2	---
1900	6.0	30
1950	18.0	40
1965	22.0	50
1975	21.6	60
1985	28.0	70
1995	28.8	80
2000*	30.0	90
2005	32.0	100
Composite Average	0.6	---

*Endpoint of service exhaustion.

TABLE 22. ANION RESIN (IRA 68) PERFORMANCE

RECAP SHEET
Exhausted with cation effluent
(per cubic foot)

Run #	NH_4OH (100%) Lbs.**	Exhaust GPM	Loading mg/l	Capacity gals.	Capacity Kgr
1	New	6	220	1878	24.1
2	3.2	6	212	1868	23.1
3	3.2	6	192	2134	23.9
4	"	"	188*	1921*	21.1*
5	"	"	212*	1794*	22.2*
Series A average (* based)			200	1858	21.7
6	3.2	4	228*	1878*	25.0*
7	"	"	212*	2008*	24.9*
8	"	"	206*	1994*	24.0*
9	"	"	240*	1731*	24.3*
10	"	"	216*	1994*	25.2*
11	"	"	240*	1808*	25.3*
Series B average (* based)			224	1902	24.8
12	3.2	2-1/3	228*	1767	23.5*
13	"	"	228*	1907	25.4*
14	"	"	206*	1897	22.9*
15	"	"	Incompleted		
16	"	"	216*	2040	25.7*
Series C average (*based)			220	1904	24.4
17	3.2	6	240*	1763*	24.8*
18	"	"	220*	1873*	24.1*
Series D average			230	1819	24.5
19	2.8	6	216*	1840*	23.3*
20	"	"	340*	1200*	23.9*
Series E average			278	1520	23.6
21	3.2	6	236*	1813*	25.0*
22	"	"	232*	1840*	24.9*
Series F average			234	1827	25.0

Table 24. Ionic breakthru from mixed bed anion resin (IRA-68) during service exhaustion.

WATER ANALYSIS
FWPCA Contract No. 14-12-599

DATE November 1970

TEST (*AS $CaCO_3$)	Raw	Carbon Filtered	Cation Effluent		Anion Effluent			
pH	7.5	8.0						
ACIDITY, MINERAL*			204.					
ALKALINITY, OH*								
ALKALINITY, CO_3*								
ALKALINITY, HCO_3*					4.	3.	2.	12.
CHLORIDES*			130.		10.	8.	10.	6.
TURBIDITY IN J.T.U.	9.0	2.5						
SULFATES*			72.		0.	0.	0.	0.
TOTAL PHOSPHATE AS PO_4	15.6	13.6	20.8		0.2	0.2	30.4	0.6
TOTAL HARDNESS*								
MAGNESIUM*								
CALCIUM*								
AMMONIA NITROGEN AS N								
NITRATE NITROGEN AS N			5.8		1.4	0.1	0.1	0.5
NITRITE NITROGEN AS N								
SODIUM*								
POTASSIUM*								
TOTAL ORGANIC CARBON	10.8	7.8	9.1		9.3	9.3	7.2	8.0
TOTAL DISSOLVED SOLIDS								
Number Gallons For 1$\frac{1}{2}$ cu ft	1053 Drip	1052 Drip	1050 Drip		1047 Start	1048 1515	1049 3010	1051 Drip IRA-68 Run #7

WATER ANALYSIS
FWPCA Contract No. 14-12-599

DATE November, 1970

TEST (*AS $CaCO_3$)								
Micromhos	30	40	50	60	70	80	90	100
ACIDITY, MINERAL*								
ALKALINITY, OH*								
ALKALINITY, CO_3*								
ALKALINITY, HCO_3*	18.	16.	12.	8.	8.	6.	6.	4.
CHLORIDES*	8.	8.	14.	10.	16.	10.	16.	20.
TURBIDITY IN J.T.U.								
SULFATES*	0.	0.	0.	0.	0.	0.	0.	0.
TOTAL PHOSPHATE AS PO_4	6.0	18.	22.	21.6	28.8	28.0	32.	32.
TOTAL HARDNESS*	0.	0.	0.	0.	0.	0.	0.	0.
MAGNESIUM*	0.	0.	0.	0.	0.	0.	0.	0.
CALCIUM*	0.	0.	0.	0.	0.	0.	0.	0.
AMMONIA NITROGEN AS N	0.2	0.3	0.4	0.3	0.2	0.3	0.3	0.3
NITRATE NITROGEN AS N	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
NITRITE NITROGEN AS N								
SODIUM*	9.6	12.	14.	15.	16.	17.	19.	19.
POTASSIUM*	0.07	0.12	0.12	0.14	0.15	0.11	0.16	0.15
TOTAL ORGANIC CARBON	7.1	6.4	6.1	6..	5.7	6.1	6.6	6.2
TOTAL DISSOLVED SOLIDS								
Number Gallons For 1$\frac{1}{2}$ cu ft	1064 2661	1065 2917	1066 2665	1067 2475	1068 2479	1069 2480	1060 3430	1061 6110

Break thru Samples IRA-68 Run #7

The regeneration of the IRA-68 has been characterized when it was regenerated with the recommended 3.2 pounds of ammonium hydroxide per cubic foot. The regenerant effluent was sampled in fractional bed volume increments. The analyses for the samples collected within the range of 2/3 to 4 bed volumes are shown on Table 24. As shown, regenerant ammonium hydroxide was initially neutralized by the adsorbed acids so that low effluent alkalinity resulted. This characteristic as well as others are much the same as they were for IRA-93 resin as shown in Table 19. The main differences between analyses for the two elutions are that phosphate elution is better with IRA-93 than it is with IRA-68; nitrate elution is also better with the IRA-93; total organic carbon is better from the IRA-93 in view of the continued elution for a longer period of time than occurs for the IRA-68.

The pilot plant studies have shown that this weak base resin IRA-68 is easily applied for demineralization of secondary treated sewage effluent. Stable operating capacities have been obtained without operational difficulties. Performance slightly favors the choice of IRA-93 over that of IRA-68 with the exception of the slightly increased capacity from IRA-68. More importantly, the capital cost for IRA-93 is about 2/3 that of the IRA-68 resin.

The recommendation is made that IRA-93 weak base resin is the choice in the selection of such a resin in combination with (and preceded by) a strong acid cation exchange resin in the hydrogen form.

TABLE 24. Regenerant effluent analyses from weak base anion resin (IRA-68). Regenerated with 3.2 lbs of 100% ammonium hydroxide.

WATER ANALYSIS

FWPCA Contract No. 14-12-599 DATE December, 1970

TEST (*AS $CaCO_3$)									
pH									
ACIDITY, MINERAL *									
ALKALINITY, OH*					2440	1660.	840.	520.	220.
ALKALINITY, CO_3*					2720.	1160.	720.	560.	440.
ALKALINITY, HCO_3*	140.	200	575	780.					
CHLORIDES*	660.	7300.	14500.	27100.	15600.	3600.	1800.	740.	480.
TURBIDITY IN J.T.U.									
SULFATES*	40.	13600.	18800.	11200.	1400.	540.	380.	260.	220.
TOTAL PHOSPHATE AS PO_4	250.	3760.	3200.	1330.	285.	85.	88.8	75.2	58.0
TOTAL HARDNESS*									
MAGNESIUM*									
CALCIUM*									
AMMONIA NITROGEN AS N	344.	9659.	11700.	12380.	6540.	1890.	1032.	550.	310.
NITRATE NITROGEN AS N	2.0	30.	60.	150.	120.	40.	32.	28.	19.
NITRITE NITROGEN AS N	0.009	0.034	0.042	0.029	0.003	0.003	0.002	0.003	0.004
SODIUM*									
POTASSIUM*									
TOTAL ORGANIC CARBON	16.7	27.4	21.8	20.4	10.0	8.0	9.2	6.2	6.2
TOTAL DISSOLVED SOLIDS									
Number	1139	1140	1141	1142	1143	1144	1145	1146	1147
Bed Volume	2/3	1	1-1/3	1-2/3	2-1/3	3	3-1/3	3-2/3	4

Regeneration Effluent IRA-68

SECTION 10

WEAK ACID CATION EXCHANGE RESIN PERFORMANCE-HYDROGEN FORM

This process employs a weak acid cation exchange resin. Rohm & Haas' resin IRC-84 was used. This resin contains carboxylic acid functional groups. Whereas strong acid cation exchange resin is capable of splitting all ionized salts in water to remove cations by an equivalent exchange for hydrogen ions, weak acid cation exchange resin can only neutralize alkaline salts. Calcium and magnesium associated with the alkalinity present in the water, are readily removed by weakly acidic resin; the capacity for sodium removal is quite low.

An example of calcium removal with this resin, represented as RCOOH, is shown in the following chemical equation employing calcium bicarbonate, one of the most typical inorganic chemicals found in water.

$$2R\text{-}COOH + Ca(HCO_3)_2 = (R\text{-}COO)_2Ca + H_2CO_3$$

Calcium is removed by an equivalent exchange for hydrogen ions from the ion exchange resin. The hydrogen ions combine with the bicarbonate ions already present in the water to form carbonic acid - H_2CO_3. The carbonic acid so formed can disassociate to form water and dissolved carbon dioxide according to the following equation.

$$H_2CO_3 = H_2O + CO_2$$

The carbon dioxide can be removed by degasification or aeration techniques.

The resin which has been exhausted tnrough the above process can be regenerated with strong acids. The following equation illustrates the regeneration with sulfuric acid.

$$(R\text{-}COO)_2Ca + H2SO4 = 2R\text{-}COOH + CaSO4$$

Sulfuric acid is normally used for regeneration because of its availability and low cost. Sulfuric acid can cause precipitation of calcium sulfate during the regeneration; therefore, care must be taken to use a low acid concentration to prevent precipitation within the ion exchange resin. If not prevented, such precipitation will result in poor performance. The precipitate will physically plug resin pores, which in turn results in reduced capacity. The precipitate will also slowly dissolve during the service cycle so that increased calcium will appear in the treated water.

Weak acid cation exchange resin will only partly demineralize waste water. The extent of demineralization will not exceed the content of alkalinity present in the water. The effluent from this resin will contain all cations associated with non-alkaline cations. Complete cation removal requires post-treatment with a strong acid cation exchange resin. If complete demineralization is not required, the weak acid cation exchange resin is the only resin process which need be applied. For example, with the sewage at Elgin, Illinois, this resin by itself reduced the inorganic salts from about 600 mg/l to about 250 mg/l. Application of the process would dictate what post-treatment methods would be required for removal of phosphate or ammonia.

Attempts to apply the hydrogen form of the weak acid cation exchange resin have been encouraging and successful. Conventional techniques of applying this resin were used. This process can be used in the design of treatment plants where only partial demineralization of the waste water is desired.

The influent water employed in the IRC-84 study was the sewage treated in the pre-treatment section of the pilot plant. This treatment consisted of dual media filtration and activated carbon adsorption.

Weak acid cation exchange resin studies were conducted in a 14" diameter tank containing 2½ cubic feet of mineral. The resin bed height was about 29" in the hydrogen form.

Regeneration was accomplished with a 0.7% sulfuric acid solution. This strength is sufficient to regenerate the resin, while avoiding precipitation of calcium sulfate. The regenerant solution was made with soft potable water as a matter of convenience. The solution was prepared in the open regenerant solution vessel, then pumped through the cation exchange resin bed. Rinse was in two stages: first with approximately 1-1/3 bed volumes of softened water at the regeneration flow rate; second, with influent service water at the service flow rate.

Observation of the recorded effluent conductivity indicated when the fast rinse was completed. Service exhaustion was then immediately initiated. Prior to this point, the conductivity decreased rapidly; during service it was stable; at exhaustion the conductivity increased. Resin capacity was calculated from the volume throughput to an effluent conductivity rise of 50 micromhos over the average (stable) conductivity during exhaustion. This exhaustion endpoint corresponds to a 10% alkalinity leakage; i.e., the effluent alkalinity was about 10% of that in the influent.

Capacity calculations were based on the above measured volume of water and its average total alkalinity. Alkalinity was determined from an analysis of the composite sample of the influent.

Table 25, "Weak Acid Cation Exchange Resin (IRC-84) Performance Summary", summarizes the performance of this resin. Four test series were run. A comparison of series A and B shows capacity is greatly affected by flow rate, which is typical for the weak acid exchange resin. At exhaustion flow rates of 6 and 3 gpm/cu ft, capacities of 17.2 and 21.5 kilograins were obtained. Effluent quality is about the same at the two flow rates. Test series C shows that greater acid utilization is obtained at a lower regenerant dosage. Test series D shows that increasing the regenerant strength and flow rate reduced the acid utilization. Series B appears to be the best system because of the lesser requirements of resin, acid, and rinse water to produce 1,000 gallons of treated water.

Table 26 recaps the individual exhaustion runs summarized in Table 25. The data has been calculated to a "per cubic foot" base, although more resin was actually used. The second column of this table shows the weight of 66° Baume sulfuric acid used for regeneration. The third column shows the exhaustion flow rate, in gallons per minute.

The loading during exhaustion, shown in the fourth column, is expressed in mg/l as calcium carbonate equivalents. The determination of this loading factor has been previously described.

The last two columns of Table 26 show the capacities obtained. The gallons shown are those delivered between the two selected start and endpoint conductivity values for the effluent as previously discussed. The capacity has been calculated, as shown in earlier sections, from the gallons produced and the loading factor to obtain the values shown in kgrs/cu ft.

The performance obtained with this resin is encouraging. Stable capacities have been fairly easily obtained, with no indication of organic fouling. Unfortunately, the methods of Rohm & Haas, the resin supplier cannot be used to predict the performance of this resin employed for sewage demineralization. Predicted capacities, as compared with the values obtained for these four test series are shown in Table 27, "Cation Resin (IRC-84) Performance-Prediction Vs Actual". The actual capacity is significantly less than that predicted.

The quality of water produced from the cation exchange resin is shown in Table 28. The table shows the typical analysis of the influent and effluent for the cation exchange resin for the four test series. As shown, the resin reduced the influent total organic carbon from a range of 6.2 - 17.6 mg/l to 6.9 - 8.9 mg/l, as measured by a Beckman Carbon Analyzer. However, analysis of samples of regeneration effluent, and resin samples removed from the test vessel, showed no evidence to substantiate this observation. The regeneration effluent does not show any elution of total organic carbon. Resin analysis does not indicate accumulation of organics.

Table 25. Weak Acid Cation Exchange Resin (IRC-84) Performance Summary, Per cubic foot base.

Test Series	A.	B.	C.	D.
Regenerant: lbs/cu ft	3.9	3.9	3.4	3.9
Strength %	0.7	0.7	0.7	1.0
gpm/cu ft	1.0	1.0	1.0	1.4
Exhaust, gpm/cu ft	6	3	3	3
Capacity, Kgr/cu ft	17.2	21.5	18.5	18.6
Acid Utilization %	66.6	83.5	93.5	71.5
Leakage, Average mg/l:				
Total hardness	23	30	54	49
Total cations	236	229	258	245
Requirements per 1000 Gallons of Water Produced:				
Cation resin, cu ft	1.22	1.00	1.15	1.19
Acid, lbs 66° Be	4.8	3.9	3.9	4.6
Rinse Water, Gals.	66	54	62	64

TABLE 26

WEAK ACID CATION EXCHANGE RESIN (IRC-84)

PERFORMANCE RECAP SHEET

PER CUBIC FOOT BASE

Run #	H_2SO_4 lb.	Exhaust gpm	Loading mg/l**	Capacity gal.	Kgr.
1	New	6	360	1066	22.4
2	3.9	6	360	1210	25.5
3	"	"	380	910	20.2
4	"	"	352	558	11.5
5	"	"	348	640	13.0
6	"	"	368	836	18.0
7	"	"	360*	827*	17.4*
8	"	"	350*	840*	17.2*
9	"	"	362	927	19.6
10	"	"	368*	790*	17.0*
Series A Average (* based)			359	819	17.2
11	3.9	3	379	1124	24.9
12	"	"	368*	1000*	21.5*
13	"	"	372*	978*	21.3*
14	"	"	366*	1026*	21.9*
15	"	"	372*	984*	21.4*
Series B Average (* based)			369	997	21.5
16	3.4	3	352*	881*	18.1*
17	"	"	368*	847*	18.3*
18	"	"	368*	885*	19.1*
Series C Average (* based)			363	871	18.5
19	3.9	3	Mechanical Difficulties		
20	"	"	372*	843*	18.4*
21	"	"	380*	847*	18.8*
22	"	"	412	843	20.3
Series D Average (* based)			376	845	18.6

** as calcium carbonate equivalent

TABLE 27

CATION RESIN (IRC-84) PERFORMANCE

PREDICTION VS ACTUAL

Test Series	A.	B.	C.	D.
Exhaust, gpm (cu ft)	6	3	3	3
Total cation influent mg/l	639	551	594	603
Hardness/ alkalinity influent	1.03	0.79	0.89	0.88
Capacity predict: Gross Kgr/cu ft	54.0	26.0	31.0	31.0
Correction for gpm	32/58	51/58	51/58	51/58
Net Kgr/cu ft	29.8	22.9	27.2	27.3
Actual Kgr/cu ft	17.2	21.5	18.5	18.6
Actual/predict capacity	58%	94%	68%	68%

Table 28 Water analyses summary of typical exhaustion of cation exchange resin (IRC-84). All influent and effluent analyses are expressed in mg/l, and as $CaCO_3$ unless specified.

Test Series	A	B	C	D
Regenerant, lb/cu ft	3.9	3.9	3.4	3.9
Exhaust, gpm/cu ft	6	3	3	3
Influent:				
Total organic carbon-C	6.2	17.6	10.9	7.4
Alkalinity	357	373	355	367
Magnesium	200	189	161	163
Calcium	167	122	154	157
Sodium	229	212	264	245
Potassium	12	16	15	20
Ammonia-N	6.4	9.4	7.9	8.6
Total non-N cations	608	539	594	585
Effluent:				
Total organic carbon-C	6.9	8.9	7.9	7.9
Alkalinity	5.3	7.3	11.0	9.0
Magnesium	17.9	15.9	26.5	28.5
Calcium	11.7	14.3	27.0	28.5
Sodium	200.0	188.0	195.0	195.0
Potassium	12.0	16.0	10.0	9.0
Ammonia-N	6.4	8.8	7.8	2.6
Total non-N cations	241.6	234.2	258.5	261.0
Reduction of cations%	60.3	56.5	56.5	55.4

A review of Table 28 shows that there is a great reduction in the calcium and magnesium content by this process. The total reduction of influent cations (exclusive of ammonia) ranges from 55.4 - 60.3%. Total influent cations were about 600 mg/l (as calcium carbonate), while the effluent total cations were about 250 mg/l. This very significant reduction is easily obtained.

Regeneration of this cation exchange resin with sulfuric acid has been without difficulty. Precipitation of calcium sulfate in the resin bed has been avoided. Specific analyses for total organic carbon elution show only analytical and sampling variation throughout the regeneration. A small excess of regenerant acid, which may require post-treatment before disposal, is present in the regeneration effluent. Neutralization of this acid is discussed in a later section of this report. The amount of neutralization required is minimal due to the high regeneration efficiency of this resin.

The recommendation is made that weak acid cation exchange resins can be applied to treat the effluent from secondary sewage treatment plants. However, weak acid cation exchange resin is only a partial means of wastewater demineralization. Many applications will require subsequent treatment or additional demineralization. Some applications may accept the effluent from this resin without futher demineralization, but selected processes may be required for removal of phosphate, which is totally unaffected by this ion exchange process, as well as for the reduction of ammonia which is only affected slightly by this resin.

SECTION 11

WEAK ACID: STRONG ACID CATION EXCHANGE RESIN

PERFORMANCE - HYDROGEN FORMS

This process employs two types of cation exchange resins: strong acid and weak acid resins. The resins are not mixed, but rather remain in discrete beds although they may be held in one pressure vessel. During exhaustion, the water to be treated passes first through the weak acid resin, then the strong acid resin. The performance of these resins to separately treat water has been previously discussed in this report.

The advantage to using both forms of cation exchange resin is the great efficiency obtained during regeneration. Excess acid is required for the regeneration of strong acid resin. This excess acid, which would otherwise require neutralization, is of sufficient quality to regenerate the weak acid exchange resin. As a result, the ion exchange capacity obtained from the weak acid exchange resin is obtained without increased operating costs. A second advantage is the reduced requirements for alkaline reagents to neutralize waste acid.

Post treatment of the effluent from these two cation resins is required to produce an acceptable water. Such treatment consists primarily of weak base anion exchange to adsorb the mineral acid produced. The weak base resin has previously been discussed in this report. Degasification is desirable to eliminate dissolved carbon dioxide from the product water. Break-point chlorination is desirable to eliminate residual ammonium nitrogen as well as for disinfection.

We have successfully applied the two types of cation exchange resins in the hydrogen form, operating in series to treat the effluent from the activated sludge treatment plant. Accepted techniques for the ion exchange industry have been applied without difficulty. It is demonstrated, therefore, that the process can be used in the design of treatment plants.

The pilot plant studies with these two resins followed two concepts. First tests were with the resins retained in separate pressure vessels. The second tests were with both resins retained in one pressure vessel, although the two resins were maintained physically separated one from the other because of their different physical properties.

The tests with resins retained in separate pressure vessels were performed with three beds of resin: a weak acid cation resin bed, and two beds of strong acid cation resin. The three beds were arranged in two systems whereby the sewage effluent flowed in parallel to a system consisting of only a bed of the strong acid exchange resin, and also

to a second system with two tanks in series--the first tank containing the weak acid resin, while the second tank contained the strong acid resin. The purpose of the parallel study on these two systems of three resin beds was to determine the merit of using two resins as opposed to using only the strong acid resin.

Table 29, "Cation Resins Summary," summarizes the performance of these three beds of cation resin. It quickly shows that the use of two resins in series is very desirable rather than using only the strong acid. Comparing the second and third columns for the two resins in series with the last column for the one resin only shows that the requirements to produce 1000 gallons of treated water is less with the two resins. Less resin (2.19 vs 3.18 cubic feet respectively); less acid (7.7 vs 9.6 pounds respectively); but more rinse water (98 vs 95 gallons respectively) are needed for the systems.

Obviously, Table 29 favors the two resin system. Operating costs can be further reduced from that suggested in the table. This is because the tests in support of Table 29 did not use waste acid for regeneration of the weak acid resin. Such acid reuse is discussed later in this section.

Tables 30, 31, and 32 are summary sheets for the individual resin beds. Table 30 is for the carboxylic (weak acid) cation resin. The data shown is "per cubic foot", although two cubic feet (with a 30" height) were actually used in the column. This resin treated the sewage effluent which had been filtered by a dual media filter in the pretreatment system. As shown, 13 cycles were made in this test series; however, run 6 through 10 were not made on the weak acid resin so that efforts were concentrated on the strong acid cation resin operating by itself. The table shows unstable capacities, which are unexplainable. However, if the four lowest capacities are considered, an average capacity of 18.7 kilograins per cubic foot were obtained from an acid regeneration dosage equivalent to 25.8 kilograins. An operational inefficiency is indicated, but this can be tolerated with regenerant reuse.

The effluent from this weak acid cation resin was then passed through a 30" bed of strong acid cation resin for additional treatment. The performance of that strong acid resin is shown in Table 31. The 13 runs for the series are again numbered on this sheet although several runs were incompleted. The last two columns in this table show the partial analysis of a composite drip sample collected from the strong acid cation resin effluent. This analysis shows the total cations (5th column) present in the average effluent from this resin. This is expressed in calcium carbonate equivalents. These cations will be delivered to service because they are sufficiently low in concentration to be acceptable.

Table 29. CATION RESINS SUMMARY

Requirements to produce 1000 gallons of treated water

Resin Type	Carboxylic only	Sulfonic after Carboxylic	Sulfonic only
Regenerant:			
lbs/cu ft	3.9	3.0	3.0
Utility, %	73	81	56
Capacity:			
Kgr/cu ft	18.7	16.0	11.0
gallons	800	1062	314
Needed/1000 gallons:			
Resin, cu ft	1.25	0.94	3.18
Acid, 66° Be	4.9	2.8	9.6
Rinse water, gal.	70	28	95

Table 30. CARBOXYLIC CATION RESIN (IRC-84) PERFORMANCE

RECAP SHEET - per cubic foot

Regenerated with 3.9 pounds 66° Be sulfuric acid, equivalent to 25.8 kilograins as $CaCO_3$. Exhausted at 3 gpm/cu ft on filtered sewage effluent.

Run #	Loading mg/l	H/A*	Capacity gal.	Kgr	Endpoint, mg/l Hardness	Alkalinity
1A	344	1.03	1535	30.9	57	32
2A	340	1.03	1000	19.9	61	42
3A	352	0.97	950	19.6	31	32
4A	328	1.09	1650	31.6	83	38
5A	333	1.15	1203	23.4	83	54
6A	Test series interrupted to concentrate efforts on other resin.					
7A	"	"	"	"	"	"
8A	"	"	"	"	"	"
9A	"	"	"	"	"	"
10A	"	"	"	"	"	"
11A	364	1.00	858	18.3	20	50
12A	364	0.89	800	17.0	13	36
13A	356	0.92	1500	31.2	11	40

* H/A refers to the ratio of influent hardness to alkalinity.

Table 31. SULFONIC CATION RESIN (IRC-120) PERFORMANCE

RECAP SHEET - per cubic foot

Regenerated with 3.0 pounds 66° Be sulfuric acid, equivalent to 19.8 kilograins as $CaCO_3$. Exhausted at 3 gpm/cu ft on carboxylic cation resin effluent.

Run #	Loading mg/l	Capacity gal.	Kgr	Composite* Cations	Ammonia
1B	Mechanical difficulties				
2B	Mechanical difficulties				
3B	312	1000	18.3	89.6	2.9
4B	274	866	13.9	118.6	2.4
5B	284	1066	17.7	44.5	2.2
6B	Test series interrupted to concentrate efforts on other resin.				
7B	"	"	"	"	"
8B	"	"	"	"	"
9B	"	"	"	"	"
10B	"	"	"	"	"
11B	252	1007	14.9	64.9	1.8
12B	264	1066	16.4	79.2	2.3
13B	256	1120	16.8	68.1	2.7

* Concentration, mg/l as $CaCO_3$, in composite sample of water treated by both cation exchange resins - carboxylic and sulfonic.

Table 32. SULFONIC CATION RESIN (IRC-120) PERFORMANCE

RECAP SHEET - per cubic foot

Regenerated with 3.0 pounds 66° Be sulfuric acid, equivalent to 19.8 kilograins as $CaCO_3$. Exhausted at 3 gpm/cu ft on filtered sewage effluent.

Run #	Loading mg/l	Capacity gal.	Kgr	Cations	Composite* Ammonia
1C	564	512	16.9	102.6	2.5
2C	584	430	14.7	82.9	2.0
3C	624	360	13.2	168.0	3.1
4C	578	283	9.6	38.1	1.6
5C	584	323	11.1	22.4	1.2
6C	520	298	9.1	17.2	0.9
7C	576	302	10.1	18.7	1.0
8C	596	312	10.8	15.4	1.7
9C	592	297	10.3	13.9	1.5
10C	588	316	10.9	16.5	1.5
11C	600	310	10.9	26.4	1.6
12C	604	313	11.0	27.5	1.3
13C	588	326	11.2	31.4	1.8

* Concentration, mg/l as $CaCO_3$, in composite sample of water treated by sulfonic acid resin only.

Table 31 also shows the ammonia concentration in the average effluent. Removal of this ammonia can be accomplished by break-point chlorination. The average capacity obtained for the last three runs on this resin was 16.0 kilograins per cubic foot.

Table 32 shows data for the 13 runs which were actually made on the 30" bed of strong acid cation resin treating the filtered sewage effluent in parallel with the above two resin beds which were operating in series. This bed of strong acid cation resin was regenerated and exhausted with the same techniques as were used on the above discussed bed of strong acid resin, with the exception that the influent waters were of different composition. The average of the last four results for the capacity with this resin was 11.0 kilograins per cubic foot. The last two columns in the table again show the cation content in the composite effluent. The cation will be delivered to service while break-point chlorination will remove the ammonia.

Exhaustion and regeneration cycles for these test series were controlled and calculated with techniques which were previously discussed in this report.

Further investigation of the applicability of using both weak and strong acid cation exchange resins was directed toward combining the resins in one pressure vessel. Such application requires that the weak acid resin must not become significantly mixed with the strong acid resin. Possible mixing is easily avoided by selecting a strong acid cation resin which is denser than the resin normally used. With this choice of resin, the strong acid resin remains below the weak acid resin. Therefore, exhaustion must be downflow for the water to contact the weak acid resin first. Similarly, regeneration with sulfuric acid must be upflow in order to avoid precipitation of calcium sulfate within the resin bed. This is accomplished because most of the calcium is removed by the upper bed of weak acid cation exchange resin. During regeneration, the eluted calcium is removed from the bed before precipitation occurs.

The denser strong acid cation exchange resin has performance characteristics which are essentially the same as the resin normally used. Preliminary studies were made with the two resins held in separate pressure vessels operated in series for separate evaluation of their performance prior to combining them in one vessel. In exhaustion, the water was first treated by the weak acid resin, then by the strong acid resin. In regeneration, the sulfuric acid was passed upflow through both resins, first through the strong acid resin then through the weak acid resin.

In combined bed approach, design considerations much primarily consider the strong acid resin, because of its operating inefficiencies. A bed heigh of 30" for the strong acid cation resin was used in our

studies. The effect of varying amounts of weak acid resin were considered.

Regeneration was with 2% sulfuric acid upflow through the strong acid resin, then through the weak acid resin. Precipitation of calcium sulfate in the regenerant effluent was found to occur about five minutes (at 80 - 100° F) after leaving the equipment.

Exhaustion was made with the effluent from the dual media filter. The exhaustion flow rate was based on four gpm per cubic foot of strong acid cation resin used. Our 30" bed required 2½ cubic feet of this resin; therefore, a total flow rate of 10 gpm during exhaustion was used in these tests. The exhaustion was monitored by means of a recording conductivity meter, positioned to test the effluent from the strong acid resin. The exhaustion was considered terminated when the effluent conductivity abruptly decreased by 500 micromhos from the stable value throughout the run. This conductivity decrease was caused by a reduction in the amount of acid produced by cation exchange.

The capacity of the individual resins was determined by methods previously described, using the volume of water treated and the loading factor. The loading factor for these studies on the two resins is the net loading factor during exhaustion, being the difference between the influent and effluent ions whose concentration was affected by the exchange resin.

Tables 33 and 34 contain the results of the exhaustion cycles performed for these tests. The tests are grouped into several series, according to the amounts of weak acid resin and regenerant acid used. Stable performance was easily obtained, so that the test values were averaged and used to prepare the summaries of Table 36. The effluent quality during these tests is shown in Table 35. The values are typical of the results obtained from the analyses of composite samples of the effluent during exhaustion. As previously discussed, this water will need post treatment to be of acceptable quality for most uses.

Table 36 summarizes the test series from Tables 33 and 34. Test series A was run with one cubic foot of weak acid resin (IRC-84) preceding (in service) 2½ cubic feet of strong acid resin (IRC-122). Utilization of the acid regenerant was 79%. An inspection of analyses made on water samples collected during the exhaustion revealed that the weak acid resin effluent contained an excessive concentration of alkaline hardness. This was not unexpected because of two factors. First, the weak acid resin bed height was only 12". Second, the exhaustion flow rate was too high: 10 gpm per cubic foot of weak acid resin. The test was made with these two conditions of poor choice to obtain a "base point" of performance for later comparison.

Table 33. Performance of two beds of cation resin in series, (IRC-84 preceding IRC-122). Counter-current flows: downflow exhaustion, upflow regeneration. Total cubic foot resin volume basis. Exhaustion at 4 gpm/cu ft of IRC-122 only.

Run No.	Cap. Gal.	IRC-84 (carboxylic) resin Cu ft 84	Load mg/l	Cap Kgr	TH/M** Infl	IRC-122 (Sulfonic) resin, 2 1/2 cu ft Load mg/l	Cap Kgr	System Capacity Kgr	Regeneration Total lbs H_2SO_4
Series A:									
3	1614	1	210	19.8	0.97	211	20.0	39.8	7.5
4	1547	1	200	18.1	0.91	248	22.5	40.6	7.5
5	1406	1	210	17.2	1.00	256	21.0	38.2	7.5
6*	1414	1	260	21.6	0.92	202	16.8	38.4	7.5
7*	1400	1	270	22.2	0.89	220	18.0	40.2	7.5
Aver.	1407	1	265	21.9	--	211	17.4	39.3	7.5
Series B:									
8	1683	1½	308	30.3	0.87	165	16.2	46.5	7.5
9	1747	1½	286	29.2	0.97	167	17.1	46.8	7.5
10	1626	1½	270	25.7	1.00	178	16.9	42.6	7.5
11*	1570	1½	254	23.3	0.97	223	20.5	43.8	7.5
12*	1648	1½	240	23.1	0.91	238	22.9	46.0	7.5
Aver.	1602	1½	247	23.2	--	230	21.7	44.9	7.5
Series C:									
13	1713	1½	270	27.0	0.90	231	23.1	50.1	8.3
14	1727	1½	260	26.3	0.88	231	23.3	49.6	8.3
15*	1572	1½	278	27.8	0.88	206	18.9	46.7	8.3
16*	1585	1½	260	24.1	0.88	238	22.1	46.2	8.3
Aver.	1578	1½	269	26.0	--	222	20.5	46.4	8.3
Series D:									
17	1625	1½	296	28.2	0.80	190	18.1	46.3	9.0
18	1737	1½	282	28.6	0.82	205	20.8	49.4	9.0
19	1666	1½	280	27.3	0.79	208	20.3	47.6	9.0
20	1973	1½	258	29.8	0.83	171	19.7	49.5	9.0
21	1926	1½	240	27.0	0.78	204	23.0	50.0	9.0
22*	1808	1½	270	28.5	0.76	232	24.5	53.0	9.0
23*	1647	1½	270	26.0	0.77	238	22.9	48.9	9.0
24*	1700	1½	290	28.8	0.77	222	22.1	50.9	9.0
25*	1718	1½	286	28.7	0.84	234	23.4	52.1	9.0
26*	1616	1½	280	26.5	0.82	261	24.7	51.2	9.0
Aver.	1698	1½	279	27.7	--	237	23.5	51.2	9.0
Series E:									
27*	1678	1½	270	26.5	0.82	250	24.5	51.0	10.0
28*	1656	1½	294	28.5	0.82	262	25.4	53.9	10.0
Aver.	1667	1½	282	27.5	0.82	256	25.0	52.5	10.0

* Data used for averages
**Ratio of influent total hardness (TH) to methyl orange alkalinity (M).

Table 34. Performance of two beds of cation resin in series, (IRC-84 preceding IRC-122). Counter-current flows: downflow exhaustion, upflow regeneration. Total cubic foot resin volume basis. Exhaustion at 4 gpm/ cu ft of IRC-122 only.

Run No.	Cap. Gal.	IRC-84 (carboxylic) resin				IRC-122 (sulfonic) resin, 2 1/2 cu ft		System Capacity	Regeneration Total lbs
		Cu ft 84	Load mg/l	Cap Kgr	TH/M** Infl	Load mg/l	Cap Kgr	Kgr	H_2SO_4
Series F:									
29*	1930	1½	312	35.3	0.82	242	27.4	62.7	11.25
30*	2027	1½	296	35.0	0.84	246	29.2	64.2	11.25
31	1968	1½	284	32.7	0.78	104	12.0	44.7	11.25
Aver.	1980	1½	304	35.2	0.83	244	28.3	63.5	11.25
Series G:									
32	2452	2	316	45.3	0.90	112	16.1	61.4	11.25
33*	2054	2	286	34.3	0.76	264	31.7	66.0	11.25
34*	2059	2	296	35.6	0.78	236	28.4	64.0	11.25
Aver.	2056	2	291	35.0	0.77	250	30.0	65.0	11.25
Series H:									
35	2204	2	276	27.6	0.81	281	37.5	65.1	12.5
36*	2501	2	298	43.6	0.81	227	33.2	76.8	12.5
37*	2388	2	302	42.2	0.81	230	32.1	74.3	12.5
38*	2174	2	314	39.9	0.81	254	32.2	72.1	12.5
Aver.	2354	2	305	41.9	0.81	237	32.5	74.4	12.5
39	2640	2	346	53.4	0.78	216	34.6	88.0	17.5
40*	3401	2	326	64.8	0.84	210	41.8	106.6	17.5
41*	3540	2	322	66.7	0.83	191	39.6	106.3	17.5
42*	3027	2	320	56.7	0.81	218	38.6	95.3	17.5
Aver.	3323	2	323	62.7	0.83	206	40.0	102.7	17.5

* Data used for averages
**Ratio of influent total hardness (TH) to methyl orange alkalinity (M).

Test series B was then run using more ($1\frac{1}{2}$ cubic feet) weak acid resin. This increased the bed height to 18 inches, while reducing the flow rate to 6-2/3 gpm per cubic foot of weak acid resin. These changes resulted in an increased regenerant utilization of 90%. Analyses of the regenerant effluent revealed that there was no mineral acidity to be neutralized at the drain. Therefore, the next four test series (C, D, E, and F) were made with progressively increasing dosages of acid regenerant. Paradoxically, waste acid was not detected in the regenerant effluent until very high regenerant dosages were used so that the utilization during exhaustion was less than 80%. With bette acid utilization, waste acid should have appeared in the regenerant effluent. This anomaly is apparently due to non-productive use of ion exchange capacity by the water used to backwash and rinse the resin during regeneration, as well as by the affect of organic ions during exhaustion and regeneration.

Reviewing the summaries for the test series C through F, it appears that the acid utilization for run E is inconsistent with the value for the other series. The likely explanation for this inconsistency is the fact that series E consisted of only two exhaustion tests, which at the time appeared to be stabilized. However, such apparently was not the case. The acid utilization for series C, D, and F was 84-85%, unaffected by the acid dosages used. Test series G, H, and I were made with an increased amount (2 cubic feet) of the weak acid resin. The acid utilization is consistent, and exceeds 85% for these three series.

Table 36 shows the amount of acid and resin required to produce 1000 gallons of treated water. These values have been used for the further presentation of cost as shown in Table 37. The following material costs were used in the calculations for Table 37.

Sulfuric acid, 66° Baume, ¢ per pound	1.6
Weak acid resin (IRC-84), $ per cubic foot	50.5
Strong acid resin (IRC-122), $ per cubic foot	23.0

The acid cost is a typical one, which will vary depending on delivery cost at the plant site. The resin costs are those which have been established by the manufacturer for industrial users of these resins.

Table 37 is best understood by inspecting Figures 15 and 16, which graph the data as a function of the amount of sulfonic acid resin used. Figure 15 graphs the data for a regenerant dosage of $7\frac{1}{2}$ pounds total of sulfuric acid, while Figure 16 refers to a regenerant dosage of $11\frac{1}{4}$ pounds of sulfuric acid. Figure 15 contains data for the use of "0% of sulfonic acid resin". This data should not be compared directly with the other data, because when no sulfonic acid resin is used the product water quality is entirely different than when sulfonic acid resin is used.

Table 35. Typical Effluent Quality From System
(Analysis in mg/l, as $CaCO_3$, of composite sample.)

Test Series	A	B	C	D
Acidity	130	140	150	140
Chloride	140	150	160	160
Sulfate	88	91	94	89
Total hardness	1.3	1.1	0.6	0.4
Ammonia-N	3.7	4.0	2.1	3.7
Sodium	106	100	96	91
Potassium	7.3	7.4	6.8	5.8
TOC-C	12	9.8	11	14.0

Test Series	E	F	G	H	I
Acidity	160	165	180	175	180
Chloride	170	170	170	165	170
Sulfate	90	91	93	91	93
Total hardness	0.4	0.3	0.3	0.1	0.4
Ammonia-N	3.2	3.4	2.6	0.9	1.9
Sodium	98.0	92.0	91.0	79.0	53.0
Potassium	6.8	5.9	5.4	4.5	2.9
TOC-C	8.8	11.0	11.0	12.0	10.0

Table 36. CATION RESINS SUMMARY

Requirements to treat filtered treated sewage

Test Series	A	B	C	D
Resin, cu ft:				
IRC-84	1	1½	1½	1½
IRC-122	2½	2½	2½	2½
Capacity:				
Gallons	1407	1602	1578	1698
Kilograirs	39.3	44.9	46.4	51.2
Acid Regenerant:				
Lb 66° Be	7.5	7.5	8.3	9.0
Kgr equivalent	50.0	50.0	55.4	60.0
% utilization	79	90	84	85
Required for 1000 gal:				
Acid, lb	5.4	4.7	5.3	5.3
Rinse, gal.	54	47	48	44
Resin, cu ft:				
IRC-84	0.71	0.94	0.95	0.89
IRC-122	1.78	1.55	1.59	1.47
Total	2.49	2.49	2.54	2.36

Test Series	E	F	G	H	I
Resin, cu ft:					
IRC-84	1½	1½	2	2	2
IRC-122	2½	2½	2½	2½	2½
Capacity:					
Gallons	1667	1980	2056	2354	3323
Kilograins	52.5	63.5	65.0	74.4	102.7
Acid Regenerant:					
Lb 66° Be	10.0	11.25	11.25	12.5	17.5
Kgr equivalent	66.7	75.0	75.0	83.3	117.0
% utilization	79	85	86	89	88
Required for 1000 gal.					
Acid, lb	6.0	5.7	5.5	5.3	5.3
Rinse, gal.	45	38	46	40	29
Resin, cu ft:					
IRC-84	0.90	0.76	0.97	0.85	0.60
IRC-122	1.50	1.26	1.22	1.06	0.75
Total	2.40	2.02	2.19	1.91	1.35

Table 37. Costs to produce 1000 gallons of cation resin treated water. Using 2½ cu ft of sulfonic acid resin with varying amounts of weak acid resin and regenerant.

Regenerant H_2SO_4, lbs:				
per cu ft 122	3	3	3	3
Total	7½	7½	7½	7½
Resin, cu ft:				
IRC-84	0	1	1½	2.2
Total	2.5	3.5	4	2.2
% IRC-122	100	71.5	62.5	0
Needed for 1000 gal:				
H_2SO_4, lb	10.92	5.4	4.7	3.9
IRC-84, cu ft	--	0.71	0.94	1.15
IR-122, cu ft	3.64	1.78	1.52	--
H_2SO_4, ¢	17.5	8.6	7.5	6.3
IRC-84, $	--	35.9	47.5	58.1
IRC-122, $	83.7	40.9	35.0	--
Resin total, $	83.7	76.8	82.5	58.1
Regenerant H_2SO_4, lbs:				
per cu ft 122	4½	4½	4½	7
Total	11¼	11¼	11¼	17½
Resin, cu ft:				
IRC-84	0	1½	2	2
Total	2.5	4	4½	4½
% IRC-122	100	62.5	55.5	55.5
Needed for 1000 gal:				
H_2SO_4, lb	16.9	5.7	5.5	5.3
IRC-84, cu ft	--	0.76	0.97	0.60
IR-122, cu ft	3.38	1.26	1.22	0.75
H_2SO_4, ¢	27.0	9.1	8.8	8.5
IRC-84, $	--	38.4	49.0	30.2
IRC-122, $	77.7	29.0	28.1	17.3
Resin total, $	77.2	67.4	77.1	47.5

Figures 15 and 16 show costs both for the regenerant acid and for the unamortized capital cost of the resin. The acid cost diminishes continually to a minimum value as the percent of sulfonic acid resin decreases. This is because as the amount of weak acid resin increases, greater utilization of the acid is achieved.

Contrary to decreased cost for regenerant acid with increased amounts of weak acid resin being used, the unamortized resin cost reaches a minimum value beyond which it increases as more weak acid resin is used. This is logical in view of the higher cost for the weak acid resin because as small amounts of the weak acid resin are added to the system "free capacity" to treat water is obtained. After sufficient weak acid resin is added to the system to deplete the excess acid, additional capacity is not obtained--the weak acid resin addition is merely wasted.

From Figures 15 and 16, it is apparent that the combined cation resin system should contain the strong acid resin in an amount equal to about 60-80% of the total cation resin. Some slight increase in the unamortized resin costs may be justifiable to achieve lower operating cost for the acid. Unfortunately, the duration of this project has been insufficient to more clearly define this area in the cost curves. Such delineation to establish optimum costs would be of interest. The final choice of the optimum ratio of the two resins can be made only after an evaluation of plant design, where total operating cost can be evaluated. Such plant design and evaluation is beyond the scope of this project. However, the choice was made to use $62\frac{1}{2}\%$ of strong acid resin in the combined cation resin bed for additional studies under this project. These studies were made to compare operating characteristics for the two cation resin system operating in a combined bed with the two resins in separate beds.

The combined resin bed was made with $1\frac{1}{2}$ cubic feet (18" bed height) of weak acid resin on top of $2\frac{1}{2}$ cubic feet (30" bed height) of strong acid resin in one vessel. Conditions used were the same as those used for the previous test series B with $1\frac{1}{2}$ cubic feet of weak acid resin and $2\frac{1}{2}$ cubic feet of strong acid resin in separate vessels, but regenerated in series with $7\frac{1}{2}$ pounds total of 66° Baume sulfuric acid.

Table 38, "Performance of Two Cation Resins in One Vessel", summarizes the test runs for the combined bed of cation resins. The loading shown in ppm is the difference in the cation content of the composite samples influent and effluent from the resin bed. The exhaustion was controlled by conductivity measurement of the effluent, as was previously discussed. The endpoint was selected when the effluent conductivity decreased 500 microhms from the stable value during service. The average performance was then used to calculate the requirements of acid and resin to treat 1000 gallons of water by the combined bed. This calculation resulted in Table 39, "Comparison of Performance", which compares the requirements to treat 1000 gallons of filtered sewage effluent with the two

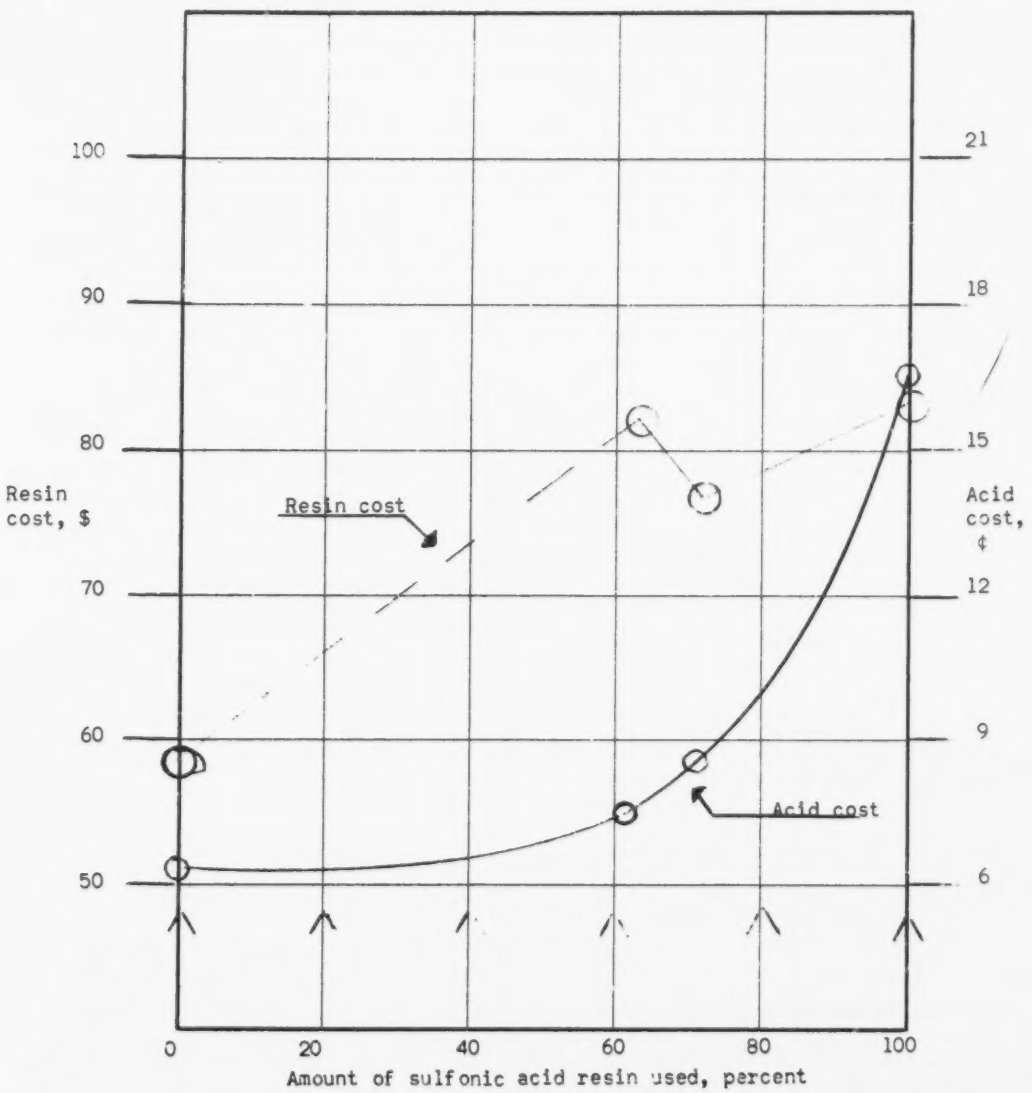

Figure 15. Costs to produce 1000 gallons of water treated by cation exchange resins. Acid costs are operating costs; resin costs are unamortized capital costs. Based on using 2½ cu ft of sulfonic acid resin regenerated with 7½ lbs of 66° Be H_2SO_4.

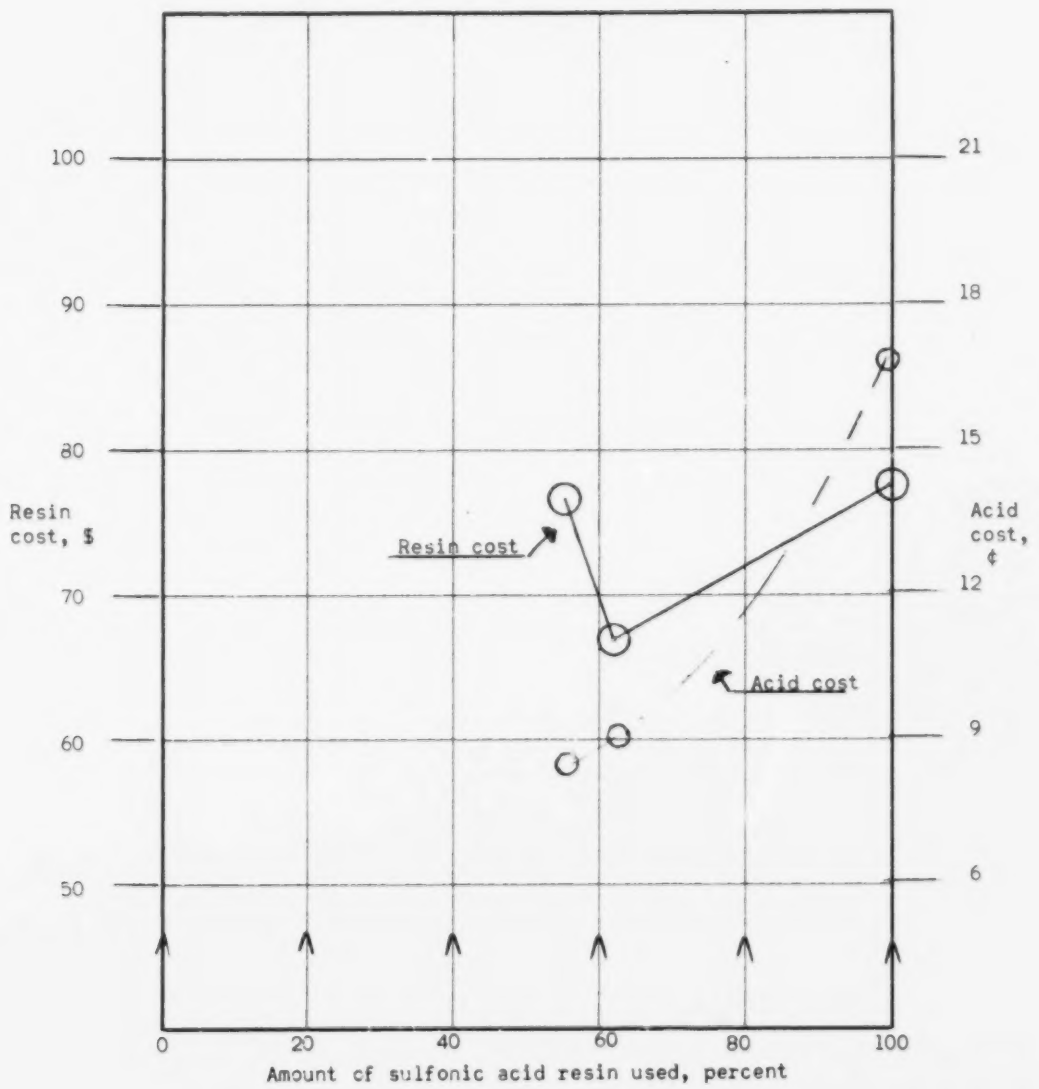

Figure 16. Costs to produce 1000 gallons of water treated by cation exchange resins. Acid costs are operating costs; resin costs are unamortized capital costs. Based on using 2½ cu ft of sulfonic acid resin regenerated with 11½ lbs of 66° Be H_2SO_4.

Table 38.
Performance of two cation resins in one vessel. Countercurrent flows: downflow exhaustion, upflow regeneration. 1½ cu ft weak acid resin on 2½ cu ft strong acid resin. Regenerated with 7½ lbs total of 66° Be H_2SO_4 as 2% solution at 2 gpm total flow.

Run No.	Loading mg/l	Capacity Gal.	Kgr.
1-S	498	1245	36.2
2-S	509	1107	33.0
3-S	495	1146	33.2
4-S	535	1279	40.0
5-S	520	1384	42.2
6-S*	555*	1388*	45.2*
7-S*	572*	1278*	42.8*
8-S		Mechanical Difficulty	
9-S*	572*	1205*	40.3*
10-S*	576*	1344*	45.2*
Average	569	1304	43.4

*Data used for averages

Table 39. COMPARISON OF PERFORMANCE

Requirements to treat filtered treated sewage with separate beds or layered beds of cation resin.

Test Series, Beds	Separate	Layered
Resin, cu ft:		
IRC-84	1½	1½
IRC-122	2½	2½
Capacity:		
Gallons	1602	1304
Kilograins	44.9	43.4
Acid Regenerant:		
Lb 66° Be	7.5	7.5
Kgr equivalent	50.0	50.0
% utilization	90	87
Required for 1000 gal:		
Acid, lb	4.7	5.8
Rinse, gal.	47	57
Resin, cu ft:		
IRC-84	0.94	1.15
IRC-122	1.55	1.91
Total	2.49	3.06
Costs for 1000 gal:		
H_2SO_4, ¢	7.5	9.3
IRC-84, $	47.5	58.2
IRC-122, $	35.0	44.0
Resin total, $	82.5	102.2

cation resins either in a separate vessel or layered into a common vessel.

Table 39 shows that the operating acid cost as well as capital resin cost are greater when the two cation resins are contained as layered beds in one vessel rather than when the resins are maintained in separate vessels. To treate 1000 gallons of water, 9.3¢ of acid are required when layered beds are used as compared with 7.5¢ when the beds are maintained separately. Also, the total unamortized resin cost is $102.20 when layered beds are used as compared with $82.50 when separate vessels are used for the resin. This disproportionate cost is sufficient to consider that layered cation beds are economically uninteresting for this process. This is in agreement with the basic premise that separate resin beds should be used when large volumes of water are being treated in order that compromises in design and performance characteristics will not be made.

The recommendation is made that both weak acid and strong acid cation exchange resins in separate beds should be applied in the demineralization of wastewater. The weak acid cation exchange resin delivers "free capacity" because this resin can be regenerated efficiently by the waste acid from the regeneration of the strong acid resin. Strong acid cation exchange resin is necessary to produce a product water with adequate quality. The effluent from these two resins is unacceptable because of the acid it contains as a result of the ion exchange process. Post treatment by means of weak base anion exchange resin to remove this acid is therefore recommended. Inclusion of aeration or degasification techniques is additionally recommended to remove the large concentration of dissolved carbon dioxide which is unaffected by the weak base anion exchange resin.

The operating material requirements for resins, regenerants, rinse water, etc. is developed in the section titled, "Operating Material Requirements For Ion Exchange Processes".

SECTION 12

WASTE REGENERANT DISPOSITION

Ion exchange resins require periodic regeneration when they become exhausted. Chemicals which are either strongly acidic or strongly alkaline are required for regeneration of cation resins or anion exchange resins respectively. Effective regeneration (to remove the exchanged ions from the resins) requires the use of regenerant in excess of the stoichiometric quantity. A large excess is required for the strong electrolyte resins, while only a small excess is needed for the weak electrolyte exchange resins.

The design of equipment to control the ion exchange processes should consider fractionation of the total effluent during regeneration and rinse. This can greatly reduce the effluent volume to be treated. Normal regeneration procedures will permit the fractionation into two portions. One of these contains most of the eluted ions and excess regenerant in a comparatively high concentration. The second portion contains the diluted rinse-out of the regenerant. This latter portion is relatively innocuous, so that disposal should be of minimal concern.

If possible, reuse of the excess waste regenerant would be very desirable for two reasons. First, reduction of disposal problems would result. Second, reduction in operating chemical costs would result.

Regenerant acid neutralization.

Acid waste from the regeneration of cation exchange resin is easily neutralized; however, precipitated sludges and neutral brines must be disposed of through locally acceptable means.

Waste regenerant acid is present in the greatest concentration and amount from the regeneration of strong acid cation exchange resin; on the other hand, a lesser concentration and amount of acid are present from the regeneration of weak acid cation exchange resin. The best use of cation exchange resin for wastewater demineralization requires the use of both strong and weak acid cation exchange resins. As discussed earlier in this report, the excess acid from the regeneration of the strong acid resin will effectively regenerate the weak acid cation exchange resin. As a consequence, the waste regenerant will require comparatively small additions of alkaline agents for neutralization.

Waste acid neutralization with alkaline agents was studied in the laboratory. Hydrated lime (calcium hydroxide) and limestone (calcium carbonate) were used in separate tests to neutralize free mineral acidity (FMA). Lime was more effective than limestone.

Conclusions were reached that lime is efficient, while a larger excess of limestone is required for neutralization. Hydrated lime dosages can be adjusted to raise the pH for complete elimination of acid, while limestone produces a maximum pH of slightly over 6. Hydrated lime can be used for neutralization of more concentrated acid, while limestone can only be used on dilute acids with a maximum FMA of about 3000 mg/l as $CaCO_3$. Economic operation requires controlled addition of hydrated lime, while limestone additions are "self-adjusting" because of its lower solubility.

Static beaker tests were separately made for acid neutralization with hydrated lime and limestone. These were run with a measured amount of analyzed excess acid being stirred in a laboratory beaker while the desired dosage of alkaline agent was added. The results can be quickly compared by inspection of the two graphs, Figures 17 and 18 which show the results respectively for hydrated lime and limestone. The graphs show that neutralization with hydrated lime is essentially complete in 10 minutes, while limestone requires about 60 minutes. The graphs show that 100% stoichiometric dosages of hydrated lime and limestone were able to raise the pH to about 2.5 and 3.5 respectively. This paradox is due to experimental error. A stoichiometric addition (quantitatively equivalent) should produce an equilibrium pH vlue which is higher than we obtained. The dosage was determined from analysis of the waste acid, and the purity of the alkaline agents. Experimental error present in these procedures resulted in a neutralizing dose that was less than stoichiometric.

Undiluted waste acid was used for the test with hydrated lime. Contrariwise, preliminary tests with limestone showed that prior dilution of waste acid was necessary for neutralization. Chemical analyses of the neutralization test solutions (before, and after addition of the required dosage) are shown in Tables 40 and 41 for lime and limestone respectively. The data supports the above conclusions.

The characteristics of the lime and the limestone used for these tests are shown in Tables 42 and 43. These tables contain information furnished by the supplier of the material.

Dynamic neutralization of the waste acid with limestone in a column was difficult. Calcium sulfate precipitation readily occured. Such precipitation can cause complete solidification of the bed into a non-reactive mass. Upflow passage of the waste acid through the limestone bed was mandatory to prevent in-place precipitation of calcium sulfate. The upflow passage of acid must be of sufficient velocity to expand the limestone bed about 50%. Such expansion provides two benefits. First, precipitates are not retained in the bed. Second, limestone particles are abraded by tumbling action so that reactive surfaces are continually exposed.

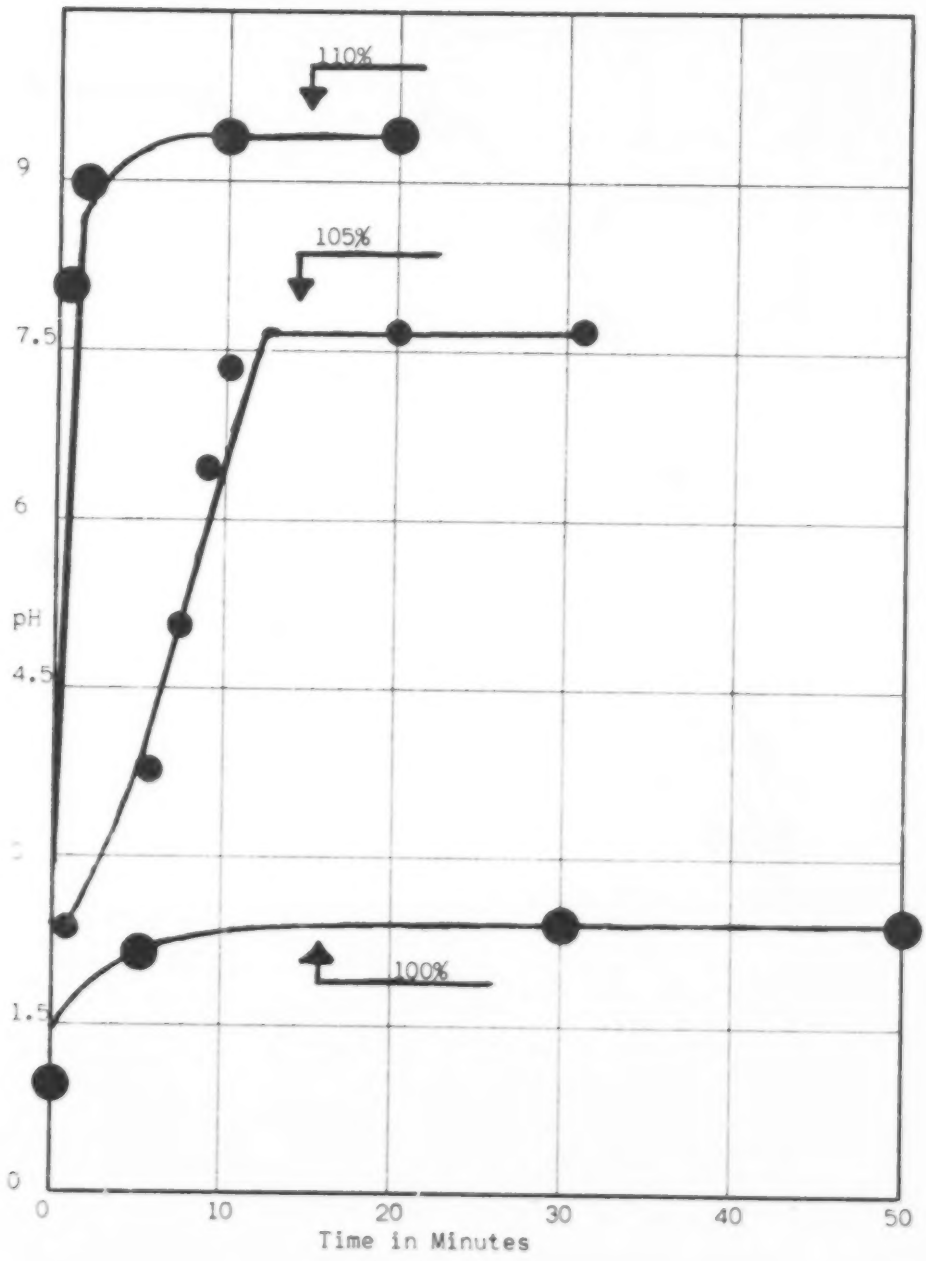

Figure 17. Static neutralization of waste acid (FMA 18900 mg/l) with 100, 105 & 110% stoichiometric lime dosage at 72° F.

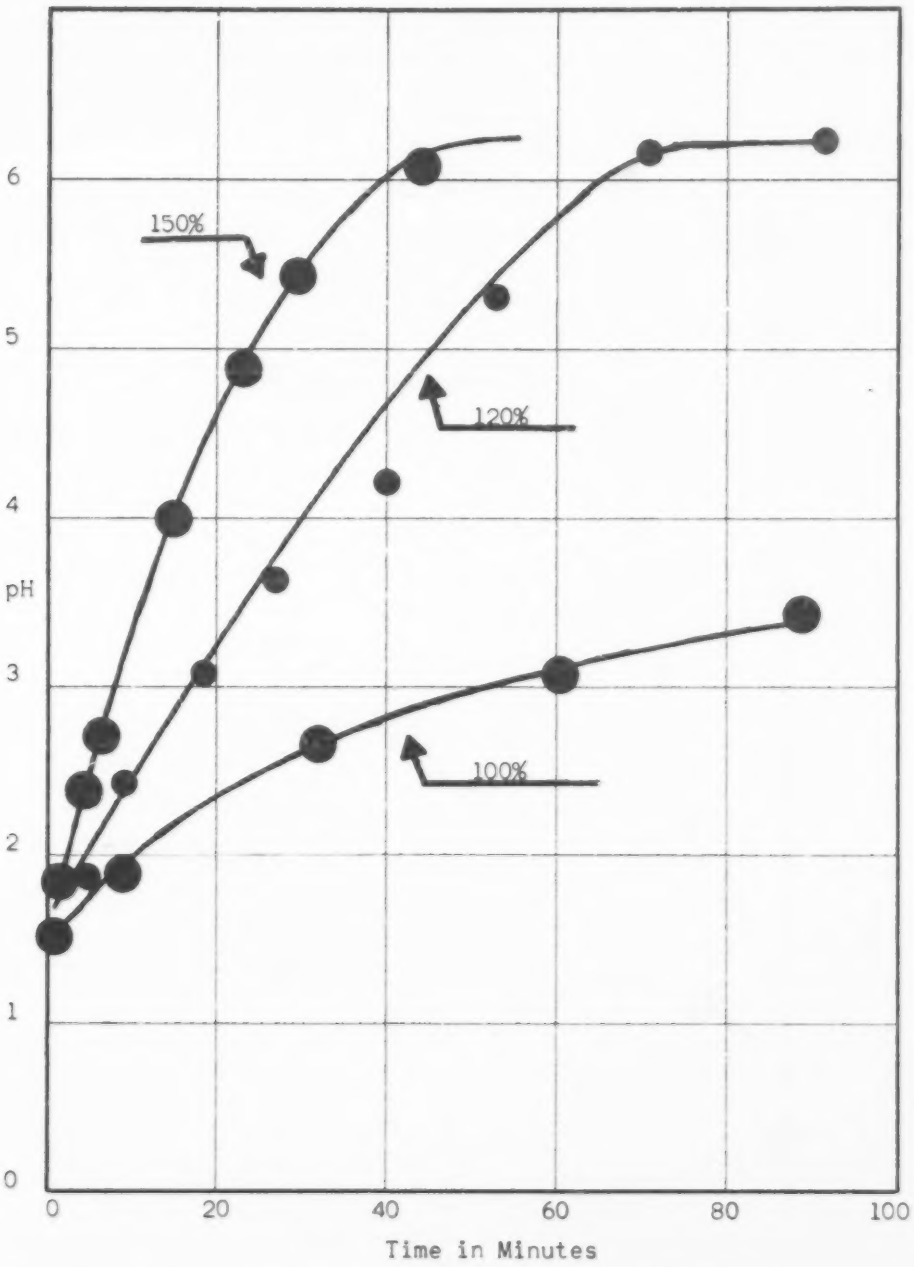

Figure 18. Static neutralization of diluted waste acid (FMA 3210 mg/l) with 100, 120 and 150% stoichiometric limestone dosages at 72° F.

Table 40

Analyses of Static Lime - Neutralized Acid Regenerant. Results expressed as grams per liter as calcium carbonate equivalents, except for pH units and sludge percent.

	Stoichiometric Dosages of Lime			
	0	100%	105%	110%
pH	1.3	2.5	8.1	9.3
Chloride	0.06	0.06	0.05	0.05
Free mineral acidity	18.90	0.70	---	---
Sulfate	35.00	16.00	15.50	13.50
Magnesium	8.00	7.40	7.50	6.40
Calcium	1.34	1.00	1.09	0.97
Sodium & Potassium	6.83	6.83	7.00	6.60
Total Alkalinity	---	---	0.10	0.38
Sludge, % by:				
Weight	0.13	2.9	6.7	7.2
Volume	---	9.0	11.8	14.2

Table 41

Analyses of Static Limestone - Neutralized Acid Regenerant. Results expressed as grams per liter as calcium carbonate equivalents, except for pH units and sludge percent.

	Stoichiometric Dosages of Limestone			
	0	100%	120%	150%
pH	1.6	3.9	6.0	6.6
Chloride	0.01	0.01	0.02	0.02
Free mineral acidity	3.21	0.01	---	---
Sulfate	6.00	4.00	3.70	3.80
Magnesium	1.28	1.35	1.34	1.38
Calcium	0.23	1.35	1.18	1.22
Sodium & Potassium	1.01	1.12	1.15	1.11
Total alkalinity	---	---	0.01	0.02
Sludge, % by:				
Weight	---	0.32	0.32	0.31
Volume	---	2.4	2.4	1.6

Table 42. Characteristics of Lime* Used

Calcium Hydroxide	97.8%
Insoluble in Hydrochloric Acid	0.01%
Chloride (Cl)	0.005%
Sulfate (SO_4)	0.05%
Iron (Fe)	0.03%
Magnesium and Alkalies (as sulfate)	1.00
Heavy metals (as Pb)	0.003
TOTAL:	98.898

* J.T. Baker #1372 (supplier analysis)

Table 43. Characteristics of Limestone Used

(For Static Test)

Typical Chemical Analysis	
Total Carbonates (Ca, Mg)	95.0%
$MgCO_3$	3.0%
Al_2O_3	0.1% to 0.25%
Fe_2O_3	0.08 to 0.19%
SiO_2	0.3% to 0.9%
Mn	Trace
Typical Physical Constants	
Hardness (Mohs' Scale)	3.0
Specific gravity	2.71
Screen Analysis	
Retained on U.S. Screen No. 16	5%
Passing U.S. Screen No. 60	10%
Acid Insolubles, by weight	1.5%

Note: All the above characteristics are issued by Georgia Marble Company (Supplier).

In addition to upflow passage to remove precipitates during limestone neutralization, such precipitates must be prevented by prior dilution of the waste acid. Figure 19 presents the results obtained from dynamic tests with limestone, made with four different dilutions of the waste acid. The diluted acid solutions were analyzed; the results appear in Table 44. As shown in Figure 19, the waste acid must be diluted to avoid neutralization slow-down.

Requirements for neutralization of waste acid are summarized as follows:

1. Use of both weak-, and strong electrolyte cation exchange resins will reduce excess acid to a minimum.

2. Regeneration controls should fractionate the effluent to reduce the volume to be treated.

3. Undiluted acid can be neutralized with hydrated lime. A dose equal to 105% of the stoichiometric requirement should be used for neutralization to a pH above 7. This should be by controlled feed of lime.

4. Dilution of waste acid to a maximum acid concentration of about 3000 mg/l (as $CaCO_3$) is necessary to neutralization with limestone. This neutralization should direct the acid upflow through an expanded bed of limestone.

5. Neutralization with lime or limestone yields a neutral saline solution and sludge, both of which require disposition by locally approved methods.

Regenerant ammonia recovery.

Waste regenerant ammonia recovery was studied in the laboratory. Regeneration of weak base anion exchange resins can be accomplished with 4% solutions of ammonium hydroxide. The effluent during regeneration will then be a solution of excess ammonium hydroxide and its various salts (chloride, sulfate, etc.). The concentration of the effluent solution will approach 4%; great dilution by rinse water can be avoided by employing fractionating techniques during regeneration. These techniques will simply direct the dilute rinse water separate from the more concentrated effluent.

The ammonium salts can be converted to ammonium hydroxide by the addition of caustic materials which will supply the necessary hydroxyl ions. The resultant ammonium hydroxide will disassociate according to the following equation to form ammonia gas and water.

$$NH_4OH = NH_3 + H_2O$$

The ammonia gas is easily volatilized from the solution.

the mixing and precipitating zones; flow being then reversed upwardly to filter through the suspended sludge blanket. The nucleation provided in the sludge blanket resulted in greater clarity through the formation of larger particles which did not rise through the upflow zone of the clarifier. At the designed 15 gallons per minute flow, the rise rate was 0.57 gallons per square feet per minute (or 4.5 ft/hour rise) at the overflow weir.

Gaseous carbonation for pH reduction was provided immediately at the effluent from the clarifier with lime clarification, this pH adjustment is necessary to prevent precipitation of scale-forming minerals in the equipment. A retention tank was provided to collect the carbonated water. This tank contained a baffle so that precipitated calcium carbonate (resulting from interaction of lime and carbon dioxide) would settle. The discharge from the baffle to the main retention zone was continually monitored by means of a recording pH meter to insure that the desired recarbonated pH value was obtained. The retention tank was a rectangular vessel holding sufficient clarified water for four hours of ion exchange system testing.

A transfer pump delivered the water from the retention tank through a totalizing water meter to the filters. This first filter was a dual media filter, 30" in diameter with a 60" side sheet. This filter contained anthracite with a effective size of 0.8 mm to a depth of 24", on top of sand with an effective size of 0.5 mm to a depth of 8".

Suitable valving was provided to permit bypass or backwash of this filter or the following carbon filter if desired. Flow indicators to verify rates were included in the plumbing.

A granular activated carbon filter was provided for adsorption of organics from the wastewater. The carbon filter was cylindrical, 54" in diameter with a 78" side sheet. It contained 80 cu ft of Pittsburgh's Filtrasorb 400 activated carbon. This was sufficient carbon to provide a contact time of 40 minutes through the empty column.

The effluent from the pretreatment system was then delivered to the ion exchange system. The pretreatment system was capable of delivering 15 gallons per minute.

The pilot plant was supplied with potable municipal water for various needs. An ion exchange water softener was provided so that water free of hardness was available for preparation of alkaline regenerant solutions.

Table 44. Analyses of Acid Waste Before Neutralization with 36" Limestone Bed

(Results in mg/l as $CaCO_3$, except pH units.)

Curve*	A.	B.	C.	D.
pH	1.45	1.6	1.6	2.1
Chloride	51	38	51	51
Free Mineral Acidity	4150	3080	3080	1280
Sulfate	4570	6080	3550	1725
Magnesium	135	1665	135	135
Calcium	230	1200	230	230
Sodium & Potassium	159	249	159	159

* See Graph #3

Hydrated lime (calcium hydroxide) is an inexpensive, readily available chemical which will supply the necessary hydroxyl ions for the above reaction. The requirements for hydrated lime to liberate the ammonia were studied under varying conditions.

The following four conditions were investigated for vacuum stripping.

1. Lime dosage, equivalent to the ammonia; 90%, 100%, and 110% of stoichiometric.

2. Temperature of the waste ammonia: 150 and 175° F.

3. Vacuum: 2.5, 10, and 20 inches of mercury vacuum.

4. Flow rate of air sweep through the system: 0.4 and 0.9 cubic feet per minute.

The waste ammonia was heated to the desired temperature, treated with the desired lime dosage, subjected to the desired vacuum and air sweep conditions. The stripped ammonia was recovered in fractions in standard solutions of acid. The recovery vessel contained methyl orange indicator for in-place titration of the ammonia. The time required to strip sufficient ammonia to neutralize the acid in the receiving flask was determined. This procedures does not permit re-use of the ammonia. The procedure was used only to study the requirements to strip the ammonia.

The effect of lime dosage on ammonia stripping is shown in Figure 20. As shown, a stoichiometric dosage of lime was adequate to recover 90% of the ammonia within 30 minutes. A slight excess of lime (10% maximum) would provide a desirable excess to insure maximum recovery.

The effect of temperature on ammonia stripping is shown in Figure 21. Elevated temperatures increased the rate of ammonia recovery. At 175°F, 90% recovery was obtained in about 30 minutes.

The effect of vacuum on ammonia stripping is shown in Figure 22. At 20 inches of mercury vacuum, 90% ammonia recovery was obtained in 30 minutes. Less vacuum drastically increased the required time.

The effect of air sweep on ammonia stripping is shown in Figure 23. Increasing the air flow rate increased the rate of ammonia recovery. The data does not show a great difference in the effect produced by the two flow rates used. The lower flow rate studied displaced in about three seconds a volume of air equal to the waste regenerant being tested. In spite of the high volume ratio for the air sweep, it was readily obtained by the vacuum source.

These vacuum stripping studies indicate that the best conditions for ammonia recovery with vacuum will include a lime dosage of 110% (of the

Conditions: 20 inches Mercury Vacuum
175° F 0.4 CFM Air Sweep

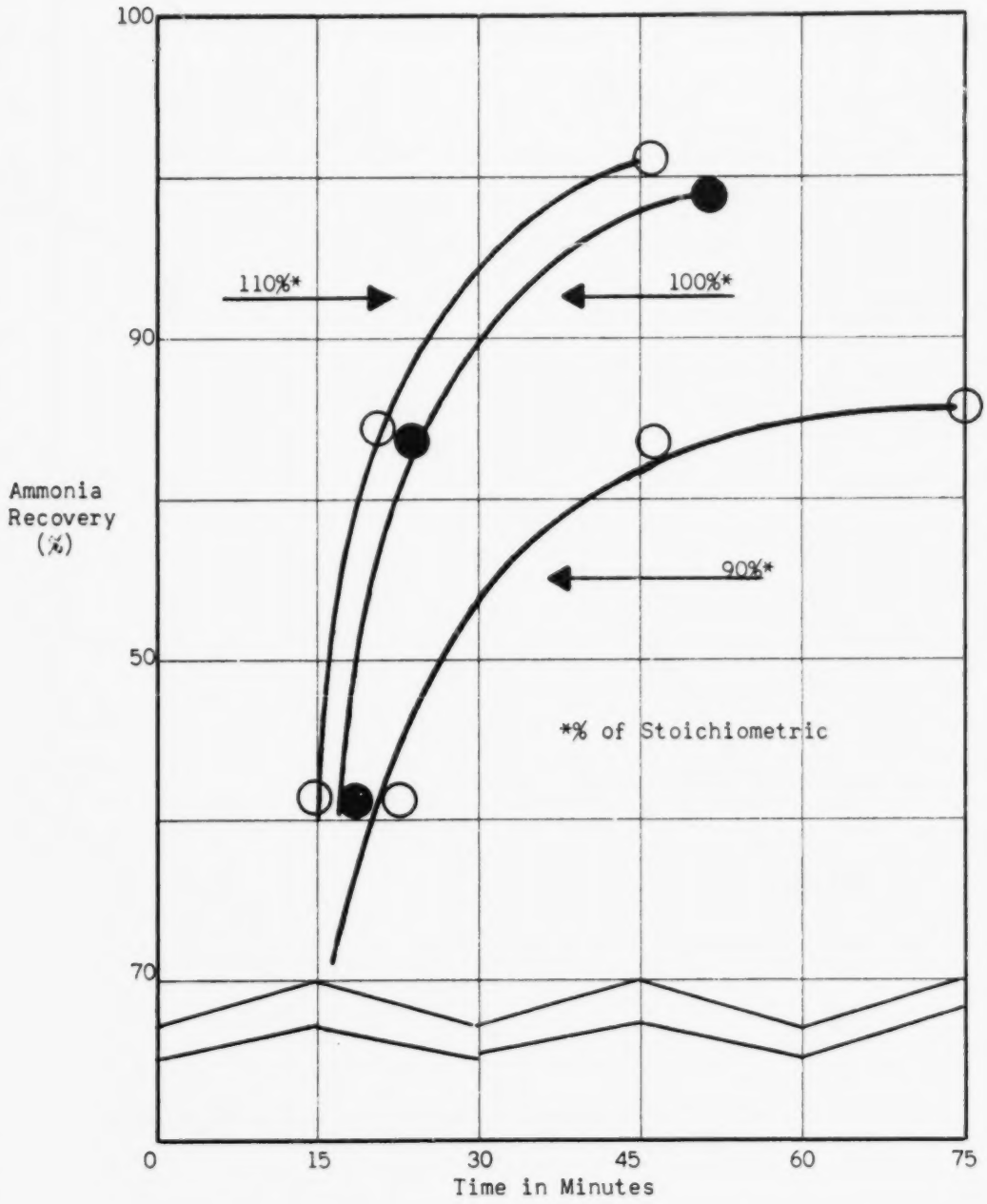

Figure 20. Effect of Lime Dosage on Ammonia Recovery

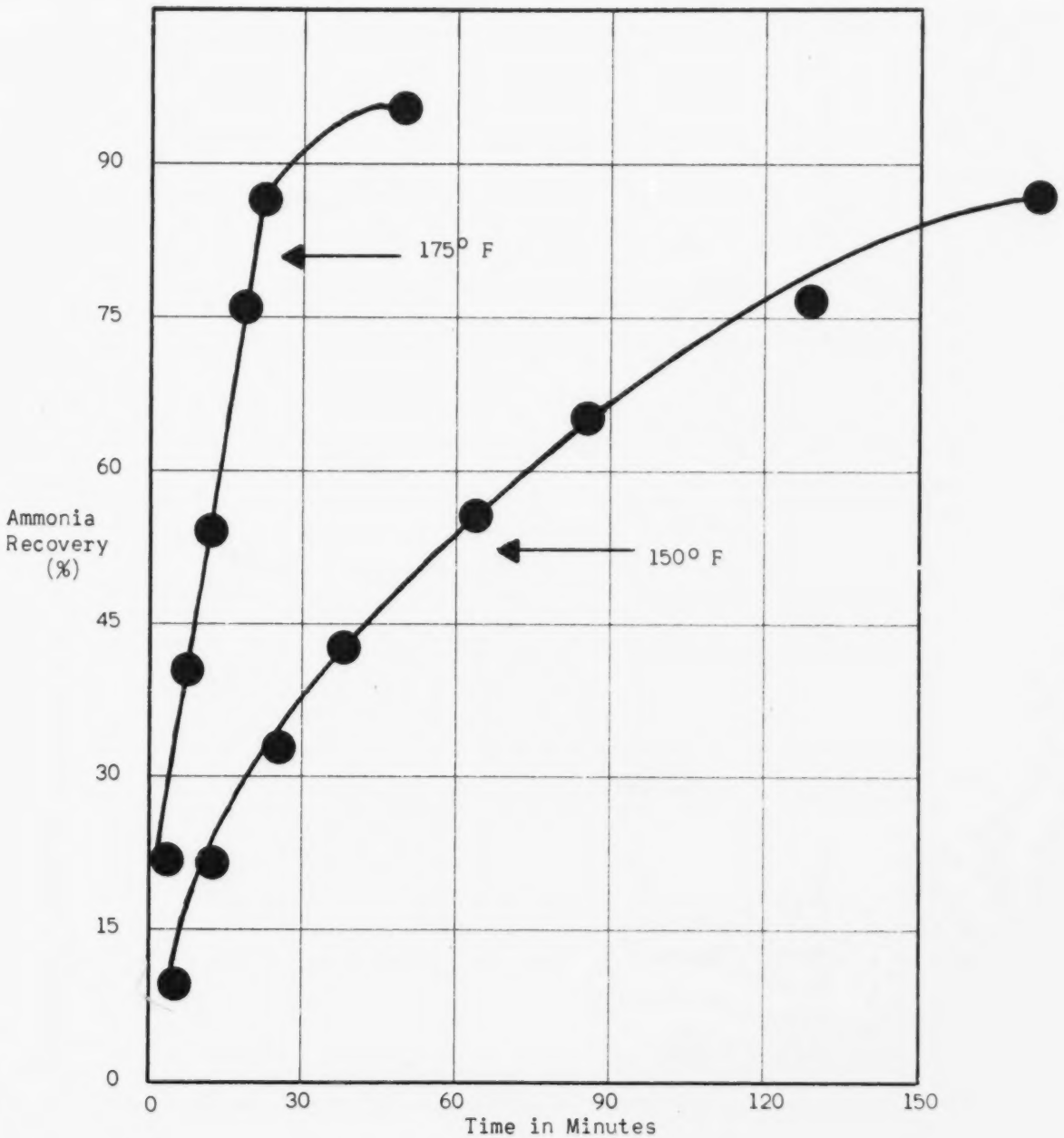

Figure 21. Effect of Temperature on Ammonia Recovery. Conditions are 20 inches Mercury Vacuum, 0.4 CFM Air Sweep, 100% Stoichiometric Lime Dosage.

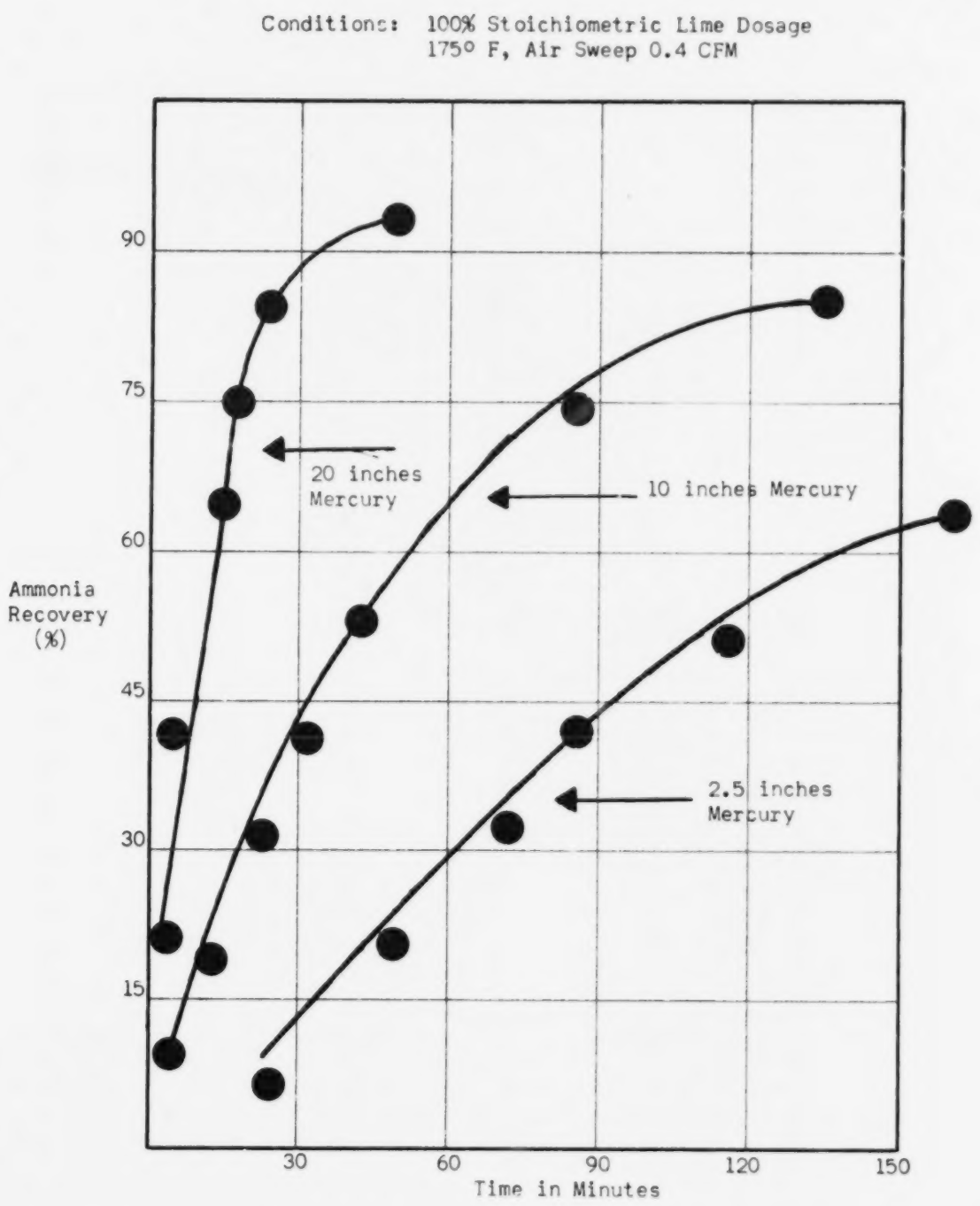

Figure 22. Effect of Vacuum on Ammonia Recovery

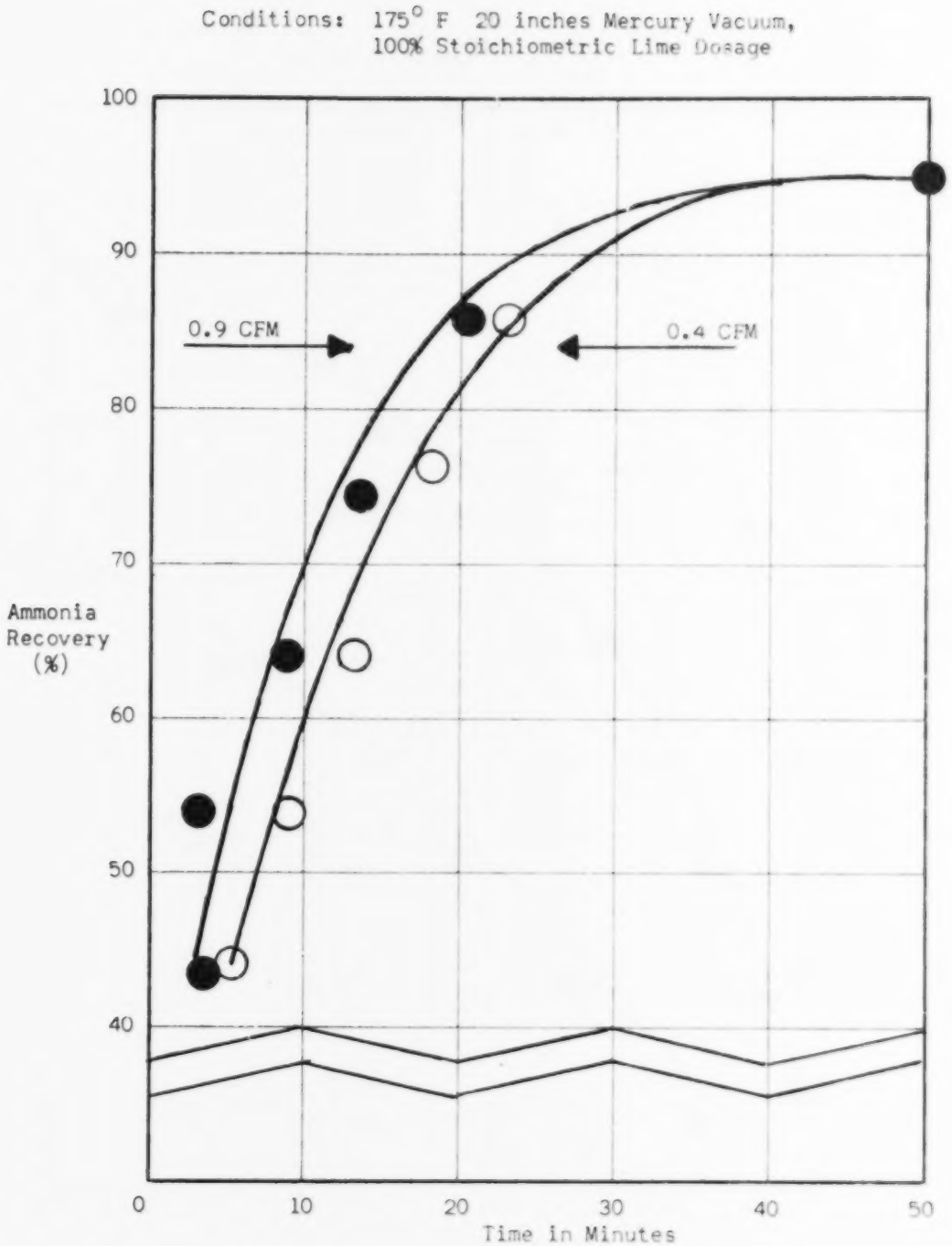

Figure 23. Effect of Air Sweep on Ammonia Recovery

stoichiometric requirement), a temperature of 175° F, 20 inches of mercury vacuum, with an air sweep. It is apparent that these conditions will be more easily obtained through direct distillation procedures.

Regenerant ammonia recovery studies included distillation. A measured quantity of waste ammonia was treated with the desired lime dosage, with steam introduced into the mixture. Ammonia and water were distilled and surface condensed into a receiving flask which contained a measured quantity of acid and methyl orange indicator. The time required to strip sufficient ammonia to neutralize the acid in the receiving flask was determined.

Two dosages of lime (100% and 120% of the stoichiometric requirement) were investigated. No significant difference was found in the effect produced by the two lime dosages. The observed data is shown in Figure 24. As shown, about 40 minutes were needed to obtain about 90% recovery of the regenerant ammonia with either lime dosage. The steam requirement to distill this ammonia was approximately 1/3 the volume of the waste ammonia solution.

It is apparent that waste ammonia is best recovered with distillation techniques. Such techniques are readily available from suppliers of distillation equipment. The recommendation of such equipment is beyond the scope of this project. The requirements for hydrated lime to liberate the ammonia are a necessary part of this project in order to predict the operating chemical cost. These costs are outlined in Section 13.

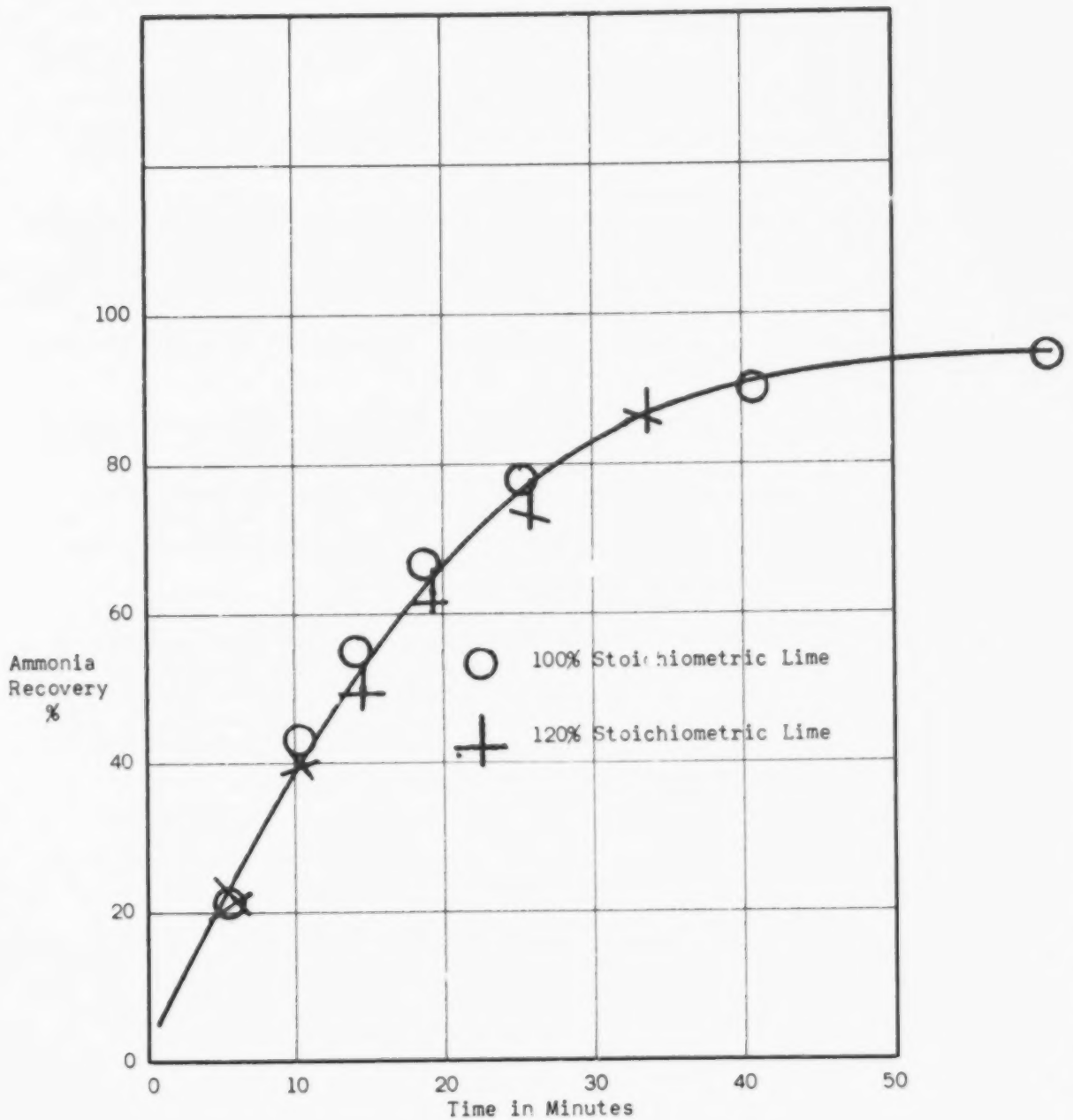

Figure 24. Ammonia Recovery by Steam Distillation.

SECTION 13

OPERATING MATERIAL REQUIREMENTS FOR ION EXCHANGE PROCESSES

Previous sections have discussed the operating performance of each ion exchange resin tested. It is the purpose of this section to list the chemical requirements to produce 1,000 gallons of treated water. Costs for equipment, labor, building, utilities, land, and other similar costs are beyond the scope of this project. The costs presented are predicated on application of the systems to treated sewage effluent of the composition which has been discussed in this report, as summarized in Table 1.

Product water costs may be varied by establishing quality standards. Better quality will increase the cost. Poorer quality should reduce the cost by blending partially treated water with the product.

Ion exchange resins must be backwashed with water before each regeneration for two reasons. First, to loosen and hydraulically classify the resin to maintain low pressure losses in the system. Second, to remove insoluble material which has been removed by filtration on the resin bed. The water used for this backwash should be clear: it can be the normal influent water to the ion exchange system. After use then, this water and its turbidity can be returned upstream in the process for clarification and reuse. The backwash water, then, is not a significant cost factor. Costs presented in this section are based on the following chemical and resin prices.

Chemical or Resin	$/Ton	¢/Pound	$/cu ft
Acid, sulfuric, 66° Be	32	1.6	--
Ammonia, anhydrous	66	3.3	--
Lime, hydrated bags	18	0.9	--
Limestone, bags	6	0.3	--
Cation resin, weak acid (IRC-120)	--	--	23.0
Cation resin, weak acid (IRC-84)	--	--	50.5
Anion resin, weak base (IRA-93)	--	--	70.5
Anion resin, weak base (IRA-68)	--	--	85.5

Strong acid cation and weak base anion, exchange resins system. (Two bed system).

This system consists of strong acid cation exchange resin (hydrogen form) followed by a weak base anion resin (free base form). Table 45 lists the requirements for operating chemicals to produce 1,000 gallons of treated water with the cation exchange resin and both (IRA-68 and IRA-93) weak base resins. The resin IRA-93 is recommended rather than IRA-68 as previously discussed.

Table 45. Chemical requirements for strong acid cation and weak base anion exchange system. To produce 1000 gallons of treated water.

Cation exchange resin (IRC-120) component:			
Regeneration level, lb/cu ft	1	3	5
Resin, cu ft	5.56	3.13	2.38
Acid, 66° Be sulfuric:			
lb	5.6	9.4	11.9
¢ (at $32/ton)	9.0	15.0	19.0
% utilization	90	53	44
Water, gallons for:			
Regenerant solution (stepwise)	34	45	46
Rinse-out	195	110	83
Waste acid (fractionated), gal.	41	54	55
Acid neutralization with:			
lime hydrated, 105% stoichiometric:			
lb	0.48	3.5	5.3
¢ (at $18/ton)	0.43	3.2	4.8
limestone, 120% stoichiometric:			
lb	0.73	5.4	8.2
¢ (at $6/ton)	0.22	1.6	2.5
Total cost, acid and lime, ¢	9.48	18.2	23.8
Anion exchange resin (IRA-93) component:			
Regeneration level lb/cu ft	1.72	2.36	2.61
Resin, cu ft	0.75	0.71	0.66
Ammonia, anhydrous:			
lb	1.29	1.68	1.72
¢ (at $66/ton)	4.3	5.5	5.7
% utilization	62	46	42
Water, gallons for:			
Regenerant solution	8	10	11
Rinse-out	65	69	75
Waste regenerant (fractionated), gal.	10	12	13
Lime, hydrated for ammonia reclaim:			
lb	1.8	1.7	1.6
¢ (at $18/ton)	1.6	1.5	1.4
Anion exchange resin (IRA-68) component:			
Regeneration level, lb/cu ft	1.72	1.96	
Resin, cu ft	0.66	0.55	
Ammonia, anhydrous:			
lb	1.13	1.08	
¢ (at $66/ton)	3.7	3.6	
% utilization	86	78	
Water, gallons for:			
Regenerant solution	7	7	
Rinse-out	34	27	
Waste regenerant (fractionated), gal.	9	9	
Lime, hydrated for ammonia reclaim:			
lb	2.1	1.8	
¢ (at $18/ton)	1.9	1.6	

Data presented in earlier sections about each resin has been abstracted into this table. Acid neutralization for the cation resin regenerant was based on the amount of unused acid and the optimum excess dosage of neutralizing agent. The total chemical costs of regenerant acid and lime neutralization ranges from 9.48 to 23.8 cents per 1,000 gallons of treated water.

Similarily shown for weak base resins are the cost for ammonia (without reclaim) and the cost of lime to release the ammonia for recovery based on a lime dosage of 105% of the stoichiometric equivalent. Thus, it is shown in the table, for IRA-93 resin, the cost to produce 1,000 gallons of treated water ranges from 4.3 to 5.7 cents for ammonia without its recovery; or, 1.6 to 1.4 cents especively for lime to recover the ammonia.

Weak acid cation exchange resin system. (One bed system.)

This system uses only one resin for demineralization to accomplish a partial reduction of ionic contamination approximately equal to the alkalinity of the influent sewage. The filtered sewage present at Elgin, Illinois and total ionized inorganic salts at a concentration of about 600 mg/l. The weak acid cation exchange resin separately was able to reduce this to about 250 mg/l. Post treatment is required, primarily for reduction of carbon dioxide, ammonia, and phosphate levels.

Table 46 lists the chemical requirements to produce 1000 gallons of treated water from only this one resin. The total chemical cost (acid plus lime for neutralization) is 6.7¢ per thousand gallons (for the partial demineralization) which can be compared to the similar total cost presented earlier of 9.48 to 23.8¢ per thousand gallons for strong acid cation resin treatment plus the cost for anion resin treatment for full demineralization.

Table 46. Chemical requirements for weak acid cation exchange system. To produce 1000 gallons of treated water.

Cation exchange resin (IRC-84) component:	
Regeneration level, lb/cu ft	3.9
Resin, cu ft	1.00
Acid, 66° Be sulfuric:	
lb	3.9
¢ (at $32/ton)	6.2
% utilization	84
Water, gallons for:	
Regenerant solution	67
Rinse-out	54
Waste acid (fractionated), gal.	80
Acid neutralization with:	
lime hydrated, 105% stoichiometric:	
lb	0.50
¢ (at $18/ton)	0.45
limestone, 120% stoichiometric:	
lb	0.77
¢ (at $6/ton)	0.23
Total cost, acid and lime, ¢	6.7

Weak acid - strong acid cation resin and weak base anion resins system. (Three bed system).

This system uses three ion exchange resins for demineralization. The system is capable of greatly reducing the concentration of all ionic contamination; however, economical operation requires continued operation until the concentration of an inorganic constituent reaches an undesirable level in the product water.

Estimated costs for this system are subject to greater variation than are the previously estimated costs. This is because the costs for the present system depend gravely upon the ratio of the amounts of the two cation resins which are used. This is because as the amount of weak acid resin increases, operating costs are decreased. Contrariwise, as the amount of weak acid resin increases, the unamortized resin cost reaches a minimum value beyond which it increases as more weak acid resin is used. It may be economically desirable to use more weak acid resin beyond this "minimum resin cost" point to achieve maximum reduction in operating costs. Selection of the optimum ratio of cation resins is beyond the scope of this project and should be clarified during detailed pilot plant studies on this system.

Table 47 shows the cost to produce 1000 gallons of demineralized water with this three bed system. For this estimate, we have used data collected for cation resin used in the ratio of $1\frac{1}{2}$ cubic feet of weak acid resin to $2\frac{1}{2}$ cubic feet of strong acid resin. A regeneration dosage of three pounds of acid per cubic foot of strong acid resin used. Unamortized resin costs were based on the resin costs established by the manufacturer for industrial users of these resins. Table 47 shows that the operating costs vary from 11.8¢ per 1000 gallons (without ammonia recovery) to 14.7¢ per 1000 gallons (with ammonia recovery). Similarily, unamortized resin costs vary from $135.5 to $129.0 respectively without and with the ammonia recovery.

The cost of the ion exchange systems are compared in Table 48.

As shown there, demineralization of wastewater at Elgin, Illinois will have a regenerant cost, per thousand gallons of product, ranging from $0.064 to $0.209. Unamortized resin costs will range from $50.50 to $135.50. The costs shown clearly indicate that the ion exchange system should use a one resin system to obtain partial reduction with economy; or, a three resin system to obtain greater reduction of salts. The two resin systems are uneconomical, comparative to the previous systems.

Table 47. Costs to produce 1000 gallons of demineralized water with three resins.

Weak acid cation resin:		
Resin, cu ft		0.94
Regenerant: reuse of that from strong acid resin		
Water, gallons for:		
Regenerant dilution		81.0
Strong acid cation resin:		
Resin, cu ft		1.52
Acid, 66° Be sulfuric:		
lbs		4.7
¢		7.5
Water, gallons for:		
Regenerant solution		28
Regenerant rinse		47.0
Weak base anion resin:		
Regeneration recovery	Without	With
Resin, cu ft	0.75	0.66
Regenerant Ammonia, 100%, lbs	1.29	1.72
Regenerant Ammonia, 100%, ¢	4.3	5.7
Lime, lbs	0	1.6
Lime, ¢	0	1.5
Water, gallons for:		
Regenerant solution	8	11
Regenerant rinse	65	75
Operating costs, ¢/1000 gal:		
Without ammonia recovery		11.8
With ammonia recovery		9.0
Unamortized resin costs:		
Without ammonia recovery:		
Cation resin, weak, $ (@ $50.5)		47.5
Cation resin, strong, $ (@ $23.0)		35.0
Anion resin, weak, $ (@ $70.5)		53.0
Total, $		135.5
With ammonia recovery:		
Cation resin, weak, $ (@ $50.5)		47.5
Cation resin, strong, $ (@ $23.0)		35.0
Anion resin, weak, $ (@ $70.5)		46.5
Total, $		129.0

Table 48. Comparison of systems to produce 1000 gallons of water by ion exchange.

System	Resin Type	Resin cu ft	Regen't lb/cu ft	Rinse gal.	Regen't cost, $	Unamort'd Resin, $
2 Resin A	Strong Cat. (IRC-120)	3.13	3.0	110	0.166*	71.990
	Weak An. (IRA-93)	0.75	1.72	65	0.043	52.875
				Total	0.209	124.865
			With ammonia recovery		0.184	124.865
2 Resin B	Strong Cat. (IRC-120)	3.13	3.0	110	0.166*	71.990
	Weak An. (IRA-68)	0.66	1.72	34	0.037	56.430
				Total	0.203	128.420
			With ammonia recovery		0.187	128.420
1 Resin	Weak Cat. (IRC-84)	1.0	3.9	54	0.064*	50.500
3 Resin	Weak Cat. (IRC-84)	0.94	Re-used	--	Nil	47.500
	Strong Cat. (IRC-120)	1.52	3.00	47	0.075	35.000
	Weak An. (IRA-93)	0.75	1.72	65	0.043	53.000
				Total	0.118	135.500
			With ammonia recovery		0.093	129.000

*Includes cost of limestone to neutralize excess acid.

SECTION 14

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SECTION 15

DEFINITIONS

ALKALINITY	Capacity to neutralize acids. In water, most alkalinity is due to the water's content of bicarbonates, carbonates, or hydroxide. The alkalinity is normally expressed in terms of calcium carbonate equivalents.
ANION	An ionic particle which is negatively charged.
BACKWASH	Reverse (normally upwards) flow through a bed of mineral or ion exchange resin to remove insoluble particulates and to loosen the bed.
BED DEPTH	The height of mineral, or ion exchange resin in a column.
BED EXPANSION	The amount of expansion given to a bed of mineral or ion exchange resin, by upflow passage of water. It is usually expressed as a percent of the unexpanded bed.
BED VOLUME	The amount of mineral, or ion exchange resin, in a column.
BREAKTHROUGH	Refers to the concentration of a particular ion, or other substance in the effluent from a treatment system. Breakthrough occurs when the effluent concentration rapidly increases. Normally, when the breakthrough concentration reaches about 10% of the influent concentration, exhaustion has occurred.

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CALCIUM CARBONATE EQUIVALENT	An expression for the concentration of constituents on a common basis for ease of calculation. Conversion of the quantity expressed "as calcium carbonate" to "as another form" requires multiplication by the ratio of the chemical equivalent weight of the desired form to that of calcium carbonate. For example, 80 mg/l of magnesium as calcium carbonate becomes 44.4 mg/l (80 x 12.2/20) as magnesium.
CAPACITY	The quantitative ability of a treatment component or system to perform. With ion exchange systems, this quantity is expressed as kilograins per cubic foot.
CATION	An ionic particle which is positively charged.
COCURRENT	Operation of a column of ion exchange resin or other mineral, with the service cycle and the regeneration cycle performed in the same direction, both either upflow or downflow.
COMPOSITE SAMPLE	A sample collected to be representative of a water flow which continues for an extended period of time.
CONDUCTIVITY	Ability of water to conduct electricity; it is the reciprocal of resistivity. Conductivity is measured in reciprocal ohms per centimeter. Water with a low concentration of ionic solids will have very low conductivity.
COUNTERCURRENT	Operation of a column of ion exchange resin or other mineral, with the service cycle and the regeneration cycle performed in opposite directions.
DEMINERALIZATION	Reduction of the ionic content of water.
DOWNFLOW	Direction of flow of solutions through ion exchange, or mineral bed columns during operation; in at the top and out at the bottom of the column.

DRIP SAMPLE	A composite sample collected by slow continuous sampling of a flowing stream.
EFFLUENT	The solution which emerges from a component or system.
ELUATE	Effluent during regeneration of an ion exchange resin. (See "Elution").
ELUENT	Influent regeneration solution to an ion exchange resin. (See "Elution").
ELUTION	The removal of an adsorbed ion or ions from an ion exchange resin during regeneration by using solutions containing relatively high concentrations of other ions. This latter solution is called the eluant. During elution, the eluant removes the adsorbed ions from the ion exchange resin: the effluent solution which contains the eluted ions is then called the eluate.
ENDPOINT	The achievement of exhaustion. With ion exchange resins, the endpoint of the service cycle is at 10% breakthrough.
EXHAUSTION CYCLE	The function of a process component in the service cycle. The regenerated form of a weak base resin without adsorbed acids.
FMA FREE MINERAL ACIDITY	Strong acids, which in water are formed principly by chloride or sulfate ions when the water has been treated by a cation exchange resin in the hydrogen form.
gpg GRAINS PER GALLON	A unit of concentration (weight per volume) that is used in the ion exchange industry. (See "GRAIN".) One gpg is numerically equal to 17.1 mg/l.
GRAIN	A unit of weight, being numerically equal to 1/7000th of a pound. (See "GRAINS PER GALLON".)

gpm	Gallons per minute.
gpm/cu ft	Gallons per minute per cubic foot of ion exchange resin or other mineral in a column.
gpm/sq ft	Gallons per minute per square foot of cross-sectional area.
HARDNESS	The sum of the calcium and magnesium ions, although other polyvalent cations are included at times. Hardness is normally expressed in terms of calcium carbonate equivalents.
ION EXCHANGE RESIN	An insoluble material which can remove ions by replacing them with an equivalent amount of a similarly charged ion.
kgr KILOGRAINS	A unit of weight (1,000 grains) equal to 1/7th of a pound.
kgr/cu ft	Kilograins (expressed as calcium carbonate) per cubic foot of ion exchange resin.
LEAKAGE	The amount of unadsorbed ion present in the effluent of a treatment component.
LIME	Lime refers to compounds of calcium. Hydrated lime is calcium hydroxide. Lime which is not hydrated is referred to as quick lime, which is calcium oxide.
MICROMHOS	Unit of measurement of electrical conductivity.
mg/l MILLIGRAMS PER LITER	A unit of concentration referring to the milligrams weight of a solute per liter of solution. The term is approximately equal to the older "part per million" term.
NEUTRALIZATION	Mutual reaction of acids and alkalies until the concentrations of hydrogen and hydroxyl ions in solution are at the desired value which is usually approximately equal.

ppm PARTS PER MILLION	A unit of concentration, which in the water treatment industry equals one part of solute in one million parts by weight of solvent. It is approximately equal to the more precise term mg/l.
REGENERANT	A solution of relatively high ionic concentration used to restore an ion exchange resin to its desired ionic form.
REGENERATION	Restoration of an ion exchange resin to its desired ionic form.
RINSE	The removal of excess regenerant from an ion exchange resin.
SALT SPLITTING	The conversion of neutral salts to their corresponding acids or bases.
SERVICE CYCLE	The use of a process component to perform its desired function.
SLUDGE	Settled precipitates of large amount.
SLUDGE BLANKET	A layer of sludge which is suspended by upflow passage of water.
SOFTENING	Removal of the hardness (calcium and magnesium ions) from water.
UPFLOW	Direction of flow of water upwardly through a component.
WEAK ACID RESIN	A cation exchange resin which cannot split neutral salts.
WEAK BASE RESIN	An anion exchange resin which cannot split neutral salts, but will merely absorb free mineral acidity.

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16. Abstract

Pilot plant studies conducted on secondary treated (activated sludge process) sewage have demonstrated the feasibility of wastewater demineralization by ion exchange.

Lime treatment to reduce phosphate is unnecessary. Filtration through dual media filters and activated carbon filters is desirable.

Partial demineralization of sewage containing a significant portion of alkaline salts is simple with one bed of weak acid (carboxylic) cation exchange resin. Complete demineralization requires at least two resins: strong acid (sulfonic) cation exchange resin and weak base anion exchange resin. The use of the weak acid cation exchange resin as a third resin will reduce operating costs and waste regenerant acid.

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17a. Descriptors

*Ion exchange, *Tertiary Treatment, *Demineralization, Acid neutralization, Activated Sludge, Activated carbon

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